METALLURGIA

THE BRITISH JOURNAL OF METALS.

INCORPORATING THE "METALLURGICAL ENGINEER."

FEBRUARY, 1950 Vol. XLI No. 244

BRITISH STEEL

Some Post-war Developments

The iron and steel industry in this country is a voluntary organisation, for the provision of common services, which is functioning under Government control with regard to steel prices and development, but the companies are financially independent and competitive units. In this form it has made tremendous progress and no industry has contributed so successfully to the need for increased production. The fear that public ownership of such a complex industry will seriously disturb this progress prompts us to give an outline of the major developments that have led to the present strong position of this organisation.

Tapping the new No. 3 blast furnace at the Clyde Iron Works of Colvilles, Ltd.

THE post-war years have been years of outstanding progress in the British iron and steel industry. During the war the industry's normal progress was hampered by more pressing production needs and many of the reorganisation schemes initiated long before the war were held up because of the time factor and labour involved in producing the new equipment needed to bring about the complete modernisation of the industry. Considerable efforts were certainly made during the war to produce more steel from the plant available and the fact that the output tonnage was so near to plant capacity showed how whole-heartedly these efforts were made by the industry. In some cases plant erections in progress at the outbreak of war were rapidly completed, and methods for increasing the output from existing furnaces, with the least possible effort, were adopted, but long-ranged schemes were of necessity held up. Thus, at the end of the war, the plant, which had been overburdened to meet abnormal demands, needed thorough overhauling, which involved modernisation and much new construction. Fortunately, the industry was alive to the production problems that would face Britain at the end of the war and, without waiting for them to accrue, prepared reorganisation schemes which could be put into operation without delay. The whole plan was submitted in the form of a Report to the Ministry of Supply and was subsequently issued as a White Paper.

The objects of the plan were: (1) To make good the further modernisation and development which would have taken place had there been no war. (2) To enlarge steel-making facilities to bring them into close relationship with the expected higher demand for steel products. (3) To ensure the most effective use of plants by concentrating production into efficient units of appropriate size, having due regard to the availability of raw materials and the distance to markets. At the rate of progress achieved during five years immediately preceding the war it was anticipated that the whole industry would be renewed by 1950. A more radical attack on the problem of rebuilding was proposed, in which full advantage was to be taken of the further technical developments which had taken place during the war. In facing this task it must be borne in mind that this very varied and complex industry was fully aware of the difficulties such a task imposed, arising from the fact that it had to be undertaken in the post-war period of re-adjustment and in a period of what was expected to be one of abnormally high construction cost.

The plan involved the building of 4,750,000 tons of blast furnace capacity and about 6,000,000 tons of steel ingot capacity. It provided for the stepping up of the rate at which new building is initiated to fully 40% of the industry's capacity in a five-year period. This exceptionally high rate envisaged was largely due to the new-construction hold-up during the war. In conjunction with the new building it was anticipated that 3,000,000 tons of the blast furnace capacity, and 4,000,000 tons of steel ingot capacity, then operating, would be scrapped. The plan as a whole had been carefully balanced to provide units of sufficient size, the most effective degree of concentration, the full loading of

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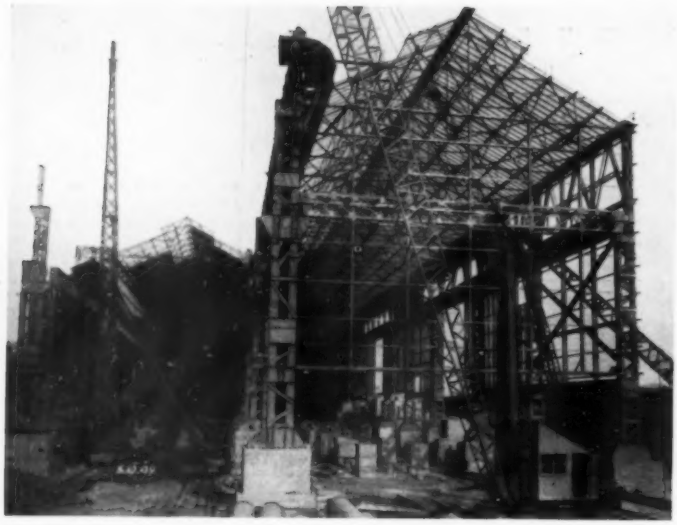


Melting shop extension in progress at the Clydebridge Works of Colvilles, Ltd.

efficient plant and the maximum reduction in fuel consumption.

The modernisation of the iron and steel industry, with a view to increased and more economical production, has been based on this plan and is still proceeding towards fulfilment. The initial expenditure contemplated was £168 million, but rising costs have increased this amount to nearer £200 million, and it should be remembered that about half of the cost of the plan is being met by the industry itself, the remainder being obtained through the usual financial channels; despite the Government control exercised over the industry, it is meeting none of this capital expenditure.

With the introduction of a new and modernised plant proceeding at a rapid rate, production has greatly increased and record outputs are frequent. So great has been the progress that Sir Ellis Hunter, President of the British Iron and Steel Federation, has sent a telegram to each of the steel ingot producers on the good start to 1950, in which he congratulates all managements and men on continuing into 1950 the increasingly fine steel ingot production records achieved by all with such consistency for so long a period. An excellent start, he said, has been made to 1950 with an annual rate of steel production of 15,873,000 tons for January. Not only has this January been the best January yet recorded, but in the last week of the month the output was the highest weekly tonnage ever achieved. In that week, the output was 328,100 tons, 5,000 tons above the previous best week.

In view of the progress made by a voluntary organisation of independent and competitive companies it is of interest to discuss briefly the development of the various schemes, completed and in progress, which are contributing so successfully to increased productivity in the iron and steel industry. For convenience the brief information is presented under the main producing areas.

The Clyde Area

Constant development, involving reconstruction and new building, is the outlook of COLVILLES, LTD. Evidence that this firm is looking to the future is seen in the many changes recorded during 1949. Among these was the return to production of No. 1 blast furnace at Clyde Iron Works; the opening of a new laboratory and the putting into operation of a new power station at Lanarkshire Steel Works; and the introduction of a chromising process at Mossend. But, while these developments are noteworthy, others of greater and far-reaching importance are the extensions now in progress at Clydebridge Works and Clyde Iron Works.

Two new blast furnaces were blown in at Clyde Iron Works in 1939, each having a capacity of 3,500 tons of pig iron per week. Apart for relining, these furnaces have since been in continuous production. The most modern furnace, No. 3, was blown in early in 1948; the assumption at the time being that one furnace should be spare, but the need for iron was such that all three furnaces are required for continuous production. The output, considerably in excess of 500,000 tons, mainly goes to the works in the Colville Group to be used in the manufacture of finished and semi-finished steel for a variety of purposes. Additional supplies of coke will come from the 68-oven battery now in process of construction and due for completion in 1952. When the scheme is completed the Clyde Iron Works will be self-sufficient in regard to coke and ample production will be available to maintain the three furnaces at their full production capacity. It is interesting to recall that more than 150 years ago pig iron was made at Tollcross, where the Clyde Iron Works now stand, and among the products of these early days were cast-iron cannon balls used by Wellington's forces to defeat Napoleon at Waterloo in 1815.

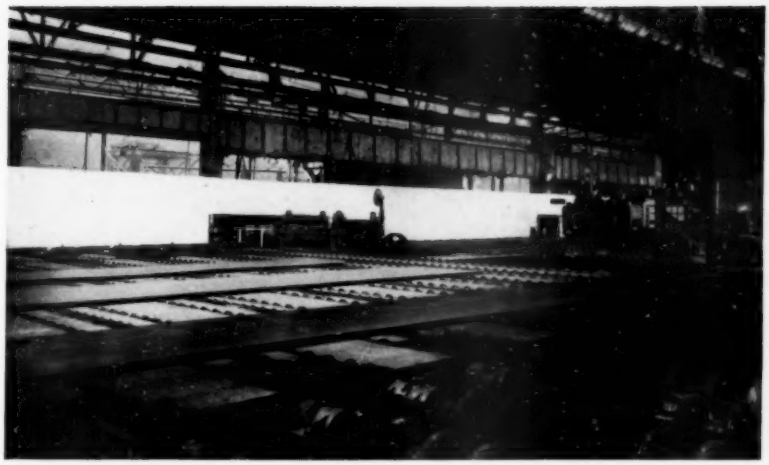
Integrated with the Clyde Iron Works are the Clydebridge Steel Works, the Company having realised that a further development of steelmaking by the hot-metal process is necessary. To further this development a substantial extension to Clydebridge Works is already well advanced. This extension comprises two additional 300-ton tilting furnaces to take liquid pig iron; the additional steel capacity from these furnaces is expected to be about 175,000 tons of ingots per annum. The furnaces, which will be completely water-cooled, in accordance with modern practice, will be fired by mixed gas from the Clyde Iron Works. Car casting will be introduced instead of the present floor casting.

In addition to the melting-shop extension, other buildings are being erected alongside for mould preparation and the other operations associated with the production of high-grade steel. New soaking pits are included in the scheme, for heating the ingots produced from the additional furnaces, as well as a large stripper bay for handling the ingots. No additions are at present contemplated to the cogging mill or to the plate mill; it is believed that, with suitable minor alterations, the present plant can handle the additional ingot production. A new slab-handling bay, however, is incorporated so that the slabs rolled at Clydebridge can be sent to other works in the Group for rolling into other finishing products.

It is anticipated that these extensions will be completed between 1950–1, and that, as each section is completed it will be put into production. When the whole scheme is in operation, Clydebridge Works will

be one of the largest and most efficient in the country.

At Lanarkshire Steel Works, Motherwell, much preparatory work has already been carried out for the change-over to larger furnace units and the entire reconstruction of the mill plant. Two of the larger furnaces are in operation and construction of a third is about to commence. These three furnaces are among the largest fixed-type open-hearth furnaces in Britain and incorporate all known features of advanced design and construction, including modern boiler plant and recovery of waste heat. Here, also, car casting will be adopted, with adequate facilities for mould preparation, stripping and quick transfer of ingots to mill. A new scrap receiving yard, now being prepared, will have all the necessary handling facilities, including a powerful bundling press for compressing the lighter classes of scrap to charging-box sizes.



A general view of the cooling banks of the recently modernised heavy plate mill at the West Hartlepool Works of South Durham Steel and Iron Co., Ltd.

At the mills, soaking pits have been rebuilt and equipped with waste-heat boilers. Considerable portions of the reconstructed blooming mill have already been completed, whilst work is proceeding in connection with its new reversing motor-drive, which is already on site. Maintenance work is carried out with the minimum of interference with current production.

North East Coast Area

Considerable development and modernisation has been in progress since 1918 at the works of the CONSETT IRON COMPANY, although intensified since 1945. Three modern blast furnaces form part of a large scheme put in hand in 1940. One of these went into operation in 1943, a second in 1947, and the third has just been completed. The furnace blown in during 1947 has beaten all production records for a furnace of its size and has produced iron with less than 14 cwt. of coke per ton. The iron produced is either transferred to a 1,000 inactive mixer, for charging the open-hearth furnaces, or is machine cast into pig iron. A modern system is applied to the handling of all raw materials for these furnaces.

To meet the needs of these blast furnaces the existing battery of 60 Wilputte coke ovens has been supplemented by a battery of 54 Woodall-Duckham Becker ovens. This battery, which at present is fired by coke-oven gas, will be heated by blast-furnace gas as soon as the new Lodge-Cottrel electrostatic gas cleaning plant, now being installed, is put into operation. With the coke-oven and blast-furnace gases, stored, respectively, in new 1 million and 2 million cub. ft. containers, the works fuel balance can be so maintained that the only coal used, apart from that used in locomotives and a small amount in the power house to overcome gas fluctuations, is that used in the coke ovens.

The output of the open-hearth furnaces have been progressively increased to over 10,000 tons per week by a series of modifications and additions to existing plant. As furnaces came off production for rebuild their capacity was brought up to 150 tons and they were fully equipped with control instruments to assist maximum production. The increased output of ingots is absorbed by the mills as a result of modernisation effected to existing soaking pits and heating furnaces and by the provision of new soaking pits and of pre-heating furnaces for ingots.

Included in the overall development is a central technical and research department, now being built, which will co-ordinate and centralise the whole of the Company's laboratory and testing work, both physical and chemical, and for routine and research. These works are now fully integrated as far as fuel and other services are concerned, and repair, traffic and research activities, on an increased scale, are being organised to secure the highest possible efficiency.

At the group of works owned by MESSRS. DORMAN LONG, developments have been proceeding to increase production from the initial materials stage right through to the fabricated products. The major projects include a new central ore unloading, preparation and sintering plant, which is now in operation. It is designed to handle 2½ million tons of ore per year and sinter 350,000 tons and to concentrate the discharge of ore for the whole group. The construction of the private rail track between Cleveland and Redcar Works is well advanced, linking the Lackenby site where the new open hearth steel plant and a universal broad flange beam mill is to be erected, with an output of 350,000 tons per annum.

The latter is the second stage of the major development plan and provides for a further capital expenditure of £8 million. The plan includes open-hearth steel plant with a capacity of 10,000 tons of ingots per week, new blast-furnace ancillary equipment and the extension of the ore grading plant already mentioned. Construction on the Lackenby site has commenced and the work is expected to be completed about September, 1952.

At the Acklam Works the old blowing engines have been replaced by modern turbo-blowers which has released additional blast-furnace gas for use at the steelworks. Increased gas cleaning facilities have been provided and two blast furnaces have been rebuilt, the hearths having been increased from 15 to 17 ft. in diameter.

Finishing end of billet mills, with Loewy shears in the foreground and the new hot banks at the Acklam Works of Dorman Long & Co., Ltd.

Modifications have been effected in the steel plant, which consists of three 250-ton tilting furnaces and a 400-ton active mixer. The furnace structures have been completely rebuilt and the water-cooling system has been improved. Since these modifications were made there has been a marked increase in furnace life and freedom from minor interruptions during a furnace campaign. The plant operates on 75% hot metal.

Improvements have been carried out to speed up and increase the normal output of the mill from 5,000 to 6,000 tons per week. The installation of a Priest bogie-type furnace has increased the ingot heating capacity. The finishing end of the mill has been completely redesigned ; new Loewy shears, fitted with automatic stopper gear and motorised measuring beam, have been installed. Complete new hot banks, and a new loading bay, fully equipped, have been constructed. The average output from these is about 6,200 tons per week.

At the Redcar plant the melting shop comprises ten fixed 85/90 ton basic open-hearth furnaces and one 400-ton active mixer. Eight of the original furnaces have been completely reconstructed to give increased bath area. During the winter of 1947 all units were converted from producer gas to fuel oil firing. The change-over took place very smoothly and it is estimated that the conversion has resulted in a 10% increased production. This steel plant is supplied with iron from two blast furnaces. During a relining programme in 1947 both furnace hearths were increased from 14½ to 18 ft. in diameter and the general furnace lines improved. As a result considerable economies were effected and individual furnace output went up from 2,000 tons per week to 3,340 tons, while coke consumption dropped to an average of 18½ cwt. per ton. These furnaces will soon be worked on a fully prepared ore burden with equivalent sinter, when further economical advantages should be obtained.

The South Durham Steel & Iron Co., Ltd. set out to modernise its steel production in 1932. As a result of the scheme a new battery of 30 Gibbons "Kogag" ovens was put into operation in 1937, and the blast-furnace plant was greatly improved in the same year. A 7 ft. 3 in. tandem plate mill was installed and commenced production in the spring of 1939. Further schemes were in active preparation for the complete electrification and modernisation of the slabbing mill and heavy plate mill, the fulfilment of which was delayed by the war, the essential machinery being obtained as soon as permits were obtained.

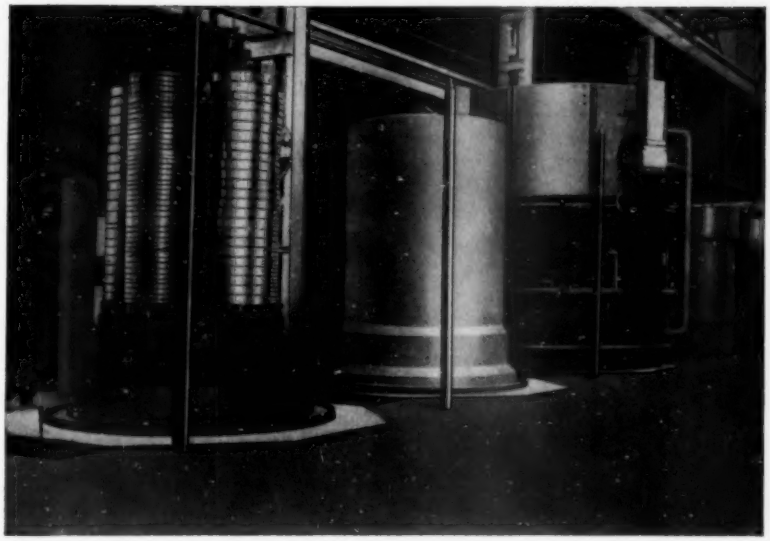
The electrification of the slabbing mill was completed early in 1946. The capacity of the mill was increased from 4,000 to 8,000 tons of slabs per week as a result. This improved supply of hot slabs had an immediate effect on the outputs of the light and heavy plate mills. The heavy plate mill was modernised, the opportunity being taken to open out the housings to enable plates up to 132 in. wide to be rolled. The scheme comprised the installation of modern working tables, a plate leveller, cooling banks and mechanical handling and shearing. This modernised mill was put into operation in the spring of 1947 and the plate finishing capacity increased from 2,000 tons to 4,000 tons per week.

Further improvements have been made at the blast-furnace plant, the output of pig iron having been increased about 50%. At the open-hearth plant improvements have also been effected. Four tilting furnaces, comprising three of 175 tons capacity and one of 90 tons capacity, have been converted to oil firing. Modifications to one of the 175-ton furnaces has resulted in 2,000 tons of ingots per week being regularly produced, operating on a ratio of 40% hot metal to 60% of scrap with an oil consumption of 24 gallons per ton of ingot. The installation of further steel furnaces has been approved and when installed the works will be self-sufficient in ingot production for obtaining a yearly production of 275,000 tons of sheared plates. The overall effect of the general improvement of plant and practice has not only resulted in an increased plate production of 2,000 tons per week but the total works fuel has been reduced from 43 cwt. per ton of finished plate to 26 cwt.

A development scheme is at present in progress at the works of Skinningrove Iron Co., Ltd. which, on completion, will increase annual output of pig iron from 180,000 tons to 310,000 tons ; ingots from 220,000 to 315,000 tons ; and rolled steel from 180,000 to 250,000 tons. Of the total pig iron output, over 50,000 tons should be available for sale. A complete new blast-furnace is being erected, together with stoves, ore bunkers, turbo-blower, and pig-casting machine. This furnace has a hearth diameter of 21 ft. and is 91 ft. high. The whole of the raw materials handling equipment for the blast-furnace plant has been redesigned. In the steel melting shop a 600-ton mixer is being erected in an extension to the north end of the existing melting shop

building. The existing active mixer is being converted into a 300-ton tilting furnace. The gas-producer plant is being rearranged and a new producer installed. Additional casting pit facilities are being provided, as is also a new electric overhead ingot stripping crane having a lifting capacity of 20 tons.

To serve the rolling mills two additional soaking pits will make seven available. In the 36-in. mill arrangements for handling material coming from the hot saw are being redesigned. A new hot bank and a heavy roller straightening machine are included. The 18-in. mill is being completely reconstructed and on completion the range of sections rolled will be increased and the output about trebled. Further installations will be two new bloom-heating furnaces and a 24-in. two-high reversing breakdown stand. The layout of the finishing end of this mill will be expanded to handle the increased output.

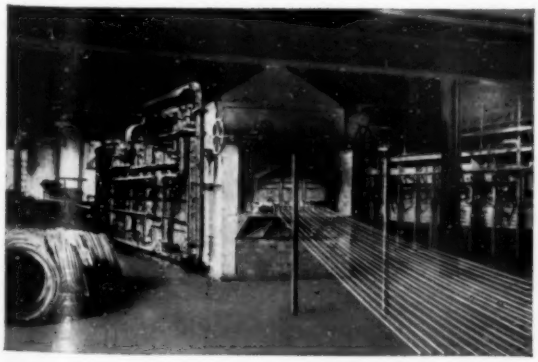


Coiled strip annealing units at Arthur Lee & Sons Ltd.

Sheffield Area

Initial development work for the manufacture of high-quality alloy steels, at the works of SAMUEL FOX & CO., LTD. of the UNITED STEEL COMPANIES, LTD., was the installation of high-frequency electric-steel melting plant in 1930. The pioneer work proved so successful that new high-frequency furnaces were installed in a new shop which also contained a duplexing furnace. With this development improvements were made to the open-hearth furnaces. The potentialities of the electric-arc furnace were investigated and in 1939 a 10-ton electric arc-melting furnace was put into operation, a further one of similar capacity being installed in 1941. Those years saw many additions to the processing plant throughout the works.

Following developments during the war a light-spring department has been established to supply high-grade coil springs, such as valve springs, for the heavy motor vehicle trade and for independent front-wheel suspension springs for motor-car manufacturers. The regular production of light clock springs in this department is also in progress. In the near future much re-planning will be carried out with the introduction of the latest equipment for processing alloy and special steels.



A wire patenting unit in operation at Arthur Lee & Sons Ltd.

The developments at STEEL PEECH TOZER'S, of the UNITED STEEL COMPANIES, LTD., have consisted largely of improvements and the modernisation of existing plants to increase outputs and obtain more economical production. All open-hearth furnaces in the Templeborough melting shop have been converted to oil firing, enabling hearths to be enlarged while rebuilding the furnaces. Additional cranes, charging machines, railways and other equipment have been installed to speed up charging. Additional ingot stripping equipment has been provided. This melting shop, with 14 furnaces, is the largest shop of its kind in Europe and has already attained an output of 572,833 tons of ingots per annum.

A new cogging mill has been installed, which, claimed to be the most efficient mill for its purpose, has attained an output, in ingot weight, of 800,000 tons per annum. It embodies all the latest improvements which enable larger outputs to be obtained at reduced costs. The two bar and strip mills have a combined output capacity of 250,000 per annum. Both are of the continuous type. Recent developments include larger heating furnaces, additional stands of rolls and other equipment. The small bar and section mills have been electrified and more heating furnaces have been installed. Buildings have been extended and more railways laid. Complete equipment for the bulk production of cold-rolled strip has been installed and is now in commission.

Considerable progress has been made in expansion and development at the various works of ENGLISH STEEL CORPORATION, LTD. At the Vicker's Works the drop forge is being modernised and extended. A large new building covering an area of 60,000 sq. ft. has been created. It is of novel design and houses the largest stamps and die-forging presses, together with ancillary machinery. New high-speed steam stamps and mechanical forging presses for die-forging, together with

The exit end of the 80-in. reversing mill at John Summers & Sons, Ltd.

The new strip galvanising furnace at John Summers & Sons, Ltd.

clipping presses and continuous furnaces, have been installed for automobile crankshafts and gears. Plant is now being installed for further induction heating and a number of new upsetting machines. Modifications are also being made to improve the efficiency of all operations in the plant.

After much research and experiment a mechanical process has been developed for the production of laminated springs, involving the use of an oil-hardening quality steel, in which sheared steel plates of suitable composition are heated to a temperature of approximately 800° C. for all the preparatory punching and blanking operations. The plates then pass to a storage tower from which they are transferred by conveyor to a continuous furnace. On leaving the furnace they are press-straightened, the plates are then formed in a specially designed press and transferred to the quenching tank, where each plate is held in accurately set dies until satisfactorily quenched. They are then transferred to a continuous-tempering furnace.

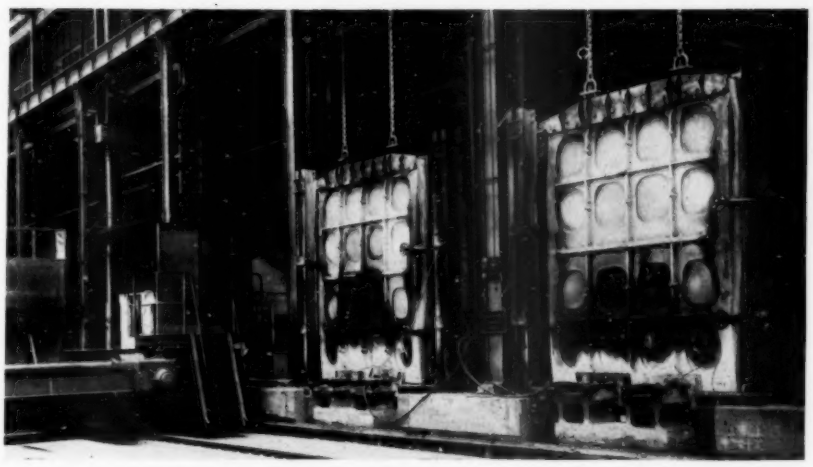
Reconstruction in the melting department has involved the installation of three 3-ton and three 1-ton capacity high-frequency induction furnaces, in which two furnaces in each group can be worked simultaneously, giving an average output of 5 tons per hour with a potential weekly output of 500 tons of special steel ingots.

Much new equipment has been installed in the machine shop during the last four years; notable additions being a heavy-duty general-purpose Craven lathe of 72 in. centres by 60 ft. length; a heavy-duty Noble & Lund lathe of 55 in. centres by 55 ft. length; a heavy-duty Wagner lathe of 69 in. centres by 72 ft. length; a Craven double-ended hollow-spindle machine of 51 in. centres, arranged to bore simultaneously from both ends or individually; and a number of other interesting machines. For dealing with very large ingots there has been installed in the heavy forge department a double-ended Noble & Lund 130-in. parting machine, capable of accommodating ingots up to 123 in. diameter by 27 ft. long. For heavy crankshaft production there is a 63-in. centres double-ended Noble & Lund lathe; while for locomotive crankshafts there are three Craven lathes.

In order to meet the demand for stainless-steel sheets, to improve their quality, and to lower production costs, FIRTH-VICKERS STAINLESS STEELS, LTD. and SAMUEL FOX & CO., LTD. in conjunction, are building a new rolling mill on a site at Shepcote Lane, Sheffield, to produce stainless-steel sheets, by the most modern methods; from cold-rolled, softened, and descaled strip up to 1 metre in width. The plant will comprise equipment for the preparation of hot-rolled slabs; hot rolling mills for rolling the slabs into strip; cold-rolling strip mills and equipment for continuously descaling both hot- and cold-rolled strip, together with the necessary ancillary and maintenance equipment.

It has been decided to install two hot reversing mills: a two-high roughing mill, with separately driven edging rolls, for the initial reductions, followed by a fast four-high finishing mill to reduce the strip to finished thickness in one heat. These hot mills, with all their auxiliary equipment, are being supplied by Davy & United Engineering Co., Ltd. For reducing the hot-rolled strip to finished thickness two cold-rolling mills are being installed. The first is of the four-high reversible type and is being built by W. H. A. Robertson & Co., Ltd. The normal function of this mill will be to reduce the hot-rolled strip to approximately 0·080 in. in thickness, and for this purpose will be used as a straight-through mill with the strip uncoiled. The second mill is of the Sendzimir cluster type, built by the Waterbury Farrel Foundry & Machine Co. The coilers, gear boxes and other mechanical auxiliaries for this mill are being made by W. H. A. Robertson & Co., Ltd., many of the parts being interchangeable with those of the four-high mill. This plant is expected to come into production during the spring of 1951.

HADFIELDS, LTD. have made steady progress with new installations and with deferred repair work in their general reorganisation scheme. The light forge has been reconstructed and equipped and is now in full production. Work on the new heavy forge is well in hand and it is expected that the plant will come into operation early next year. Among the forging presses to be installed are a 2,700-ton air hydraulic press and a 1,500-ton hydraulic press, both of which are being supplied by Loewy Engineering Co., Ltd. Nine bogie-hearth furnaces, five fixed-hearth furnaces, and three re-circulating type heat-treatment furnaces are also being installed.



Two of the new dead end annealing furnaces installed at John Summers & Sons, Ltd.

In the steel foundries existing steelmaking furnaces are to be replaced by a side-blown converter plant comprising two converters with provision for a third. The converter plant is expected to be in operation during the autumn of this year. In addition a 15-ton electric-arc furnace and two 1-ton high-frequency furnaces, complete with equipment, will be included. A fully mechanised foundry plant is to be installed.

In the 28-in. mill two reheating furnaces have been reconstructed which has resulted in a marked increase in the capacity of the mill. In addition a new ingot charger is to be installed. At the open-hearth furnaces the existing gas-producer plant has been modernised. Generally the whole reorganisation scheme provides for a considerable number of new machines and the re-siting of existing ones to enable the shops to be operated on an efficient flow-line system.

During the war ARTHUR LEE & SONS, LTD. assisted very considerably in meeting the demand for cold-rolled strip. Early in 1943 very thin gauges were needed, such as 0·004 and 0·003 in. and even as low as 0·002 in., which called for immediate action and a scheme was put into operation which enabled economical and efficient production in sufficient quantities. The scheme involved two new shops and new plant. With the end of the war the production of stainless-steel strip, which had been considered as a development in 1939, was reviewed, as a result of which the directors developed a five-year plan to develop the production of stainless-steel strip.

Existing cold-rolling plant not being entirely suited for the production of stainless-steel strip a Sendzimir mill was installed capable of rolling stainless-steel strip from a hot roll size of about $\frac{1}{8}$ in. thick to any thickness desired, without intermediate annealing. In order to provide strip of a very high finish, Hamilton surface grinders are to be used. With these grinders a larger coil in its finished state is unwound and passed through a battery of these machines which grind or polish the surface to a precise degree, as required, and subsequently rewind the coil.

This Company, in addition to making a speciality of stainless-steel strip, make various qualities of steel strip, wire, bars, etc., but the stainless-steel strip project is an outstanding one.

The products of THE PARK GATE IRON & STEEL CO., LTD. are now confined to billets, bars, and sections. Production has expanded during the war years as a result of plant modifications. At present the plant consists of two blast furnaces and nine open-hearth basic furnaces. There is a combined blooming and billeting mill and 10, 12, 18 and 24-in. finishing mills. All the open-hearth furnaces were converted to oil firing by the beginning of 1948, which not only made a contribution to increased production, but has been an important factor in a 25% increase in the fuel efficiency of the works.

To keep in step with increased ingot production it was necessary to increase the rate of ingot rolling. To achieve this, the following structural alterations have been made. The slabbing mill has been converted for blooming the ingots, with facilities for transferring the blooms to the billeting stand. The controls of both blooming and billeting mills have been modernised. Electrically-driven Lake-rotating manipulators have been installed at the billeting mill. The run-out on the delivery side from the billeting rolls has been lengthened. Equipment for transferring the sheared billets to an adjacent bay has been installed. Changes have also been effected in the finishing mills.

Further developments in progress include the conversion of the blast-furnace plant to mechanical handling of raw materials. This involves the installation of two wagon tipplers and the conveying of the materials on belt conveyors to storage bunkers. Materials will be collected from the bunkers in scale cars and conveyed to the hoists. The operations at the furnaces will be fully mechanical and the charging of the furnaces under automatic control. The scheme also provides for electric blowers to replace the existing gas engines. Revision of, and addition to, the scrap handling facilities for the steelmaking plant has just been completed, in connection with which a new crane and a charging machine have been installed. Additional to the present rolling mill plant an ingot reheating furnace is to be installed; roller gear in the blooming mill will be revised; mechanical handling equipment on the cogging mill hot bank has been extended; a number of mill reheating furnaces have been converted to oil fuel; and a revision of the hot bank in the 12-in. mill is contemplated. An 11-in. continuous bar mill is also to be installed. When the whole of the programme is complete, production at

Aerial view of Round Oaks Steel Works Ltd. showing new works.

these works is expected to average 3,000 tons of pig iron and 7,000 tons of basic steel ingots per week.

North West Area

The first major re-organisation of the Irlam Works of the LANCASHIRE STEEL CORPORATION was carried out between the years 1930 and 1933 and resulted in the present layout. In 1944 the rate of throughput of the mills was increased by the installation of a new 36-in. semi-cogging mill, a 34-in. general roughing mill and a 32-in. finishing stand. In 1947 a second 32-in. finishing stand was introduced. Modern soaking pits, mostly with full automatic control, were designed and introduced to ensure correct rolling temperatures for the steel, reduce scale and slag formation, and give minimum gas consumption per ton of ingots.

During the secondary period of development every opportunity has been taken of increasing outputs and plant operating efficiencies by the installation of modern instruments and automatic control equipment. Further reorganisation and development has been put in hand with a view to reducing operating costs and increasing outputs. The present output of pig iron is 4,000 tons per week and of steel from the open-hearth plant, 7,500 to 8,000 tons per week.

In their reorganisation and development scheme JOHN SUMMERS & SONS, LTD. have installed the following plant since 1945: A three-zone pusher type slab furnace, with a nominal capacity of 50 tons of slab per hour, was put into commission in March, 1946. Their original oil-fired "L" open-hearth furnace commenced operations in May, 1946. This was one of the pioneer oil-fired open-hearth furnaces and many furnaces in other works were subsequently converted on similar lines. Three additional furnaces were later converted to oil firing by this Company. An 80-in. reversing cold reduction mill went into production in September, 1946. A building for a new annealing plant was completed in August, 1947, and the existing annealing plant transferred to the new building. Subsequently a new annealing plant was installed and was in full production about 12 months later. These furnaces are arranged for either sheet or coil annealing. Four new oil-fired soaking pits, designed by the Company, were completed in 1947 and all went into production before the end of that year. Two slab furnaces were converted to oil firing the same year. An 80-in. temper pass mill, a 4-high unit, was installed in August, 1948. It is a double-purpose mill and can be arranged to temper pass steel in either sheet or coil form. An additional stand to the existing 3-stand tandem mill, together with ancillary gear, was also installed in August, 1948, while a few months later an 80-in. coil slitting and trimming line unit was installed and early in 1949 a 74-in. rotary shear line unit went into production for side trimming of steel in coil form and for shearing into sheet lengths. The first section of the converted annealing plant went into production about March, 1949, and the whole job was completed by the end of August, 1949. New equipment included a Churchill roll grinder, installed last year, and two shot-blast plants.

Considerable developments are at present in progress at these Chester works. The plant under construction including a 42-in. slab mill with a lift of 61 in.; a complete steelmaking plant comprising raw materials bay and melting shop proper for the production of steel up to the ingot stage, with eight 150-ton fixed open-hearth furnaces. A complete blast-furnace plant is being laid out, which will comprise two 27-ft. blast furnaces; a complete coke-oven plant is also included, comprising 88 ovens with provision for expansion to 132 ovens and with all ancillary plant.

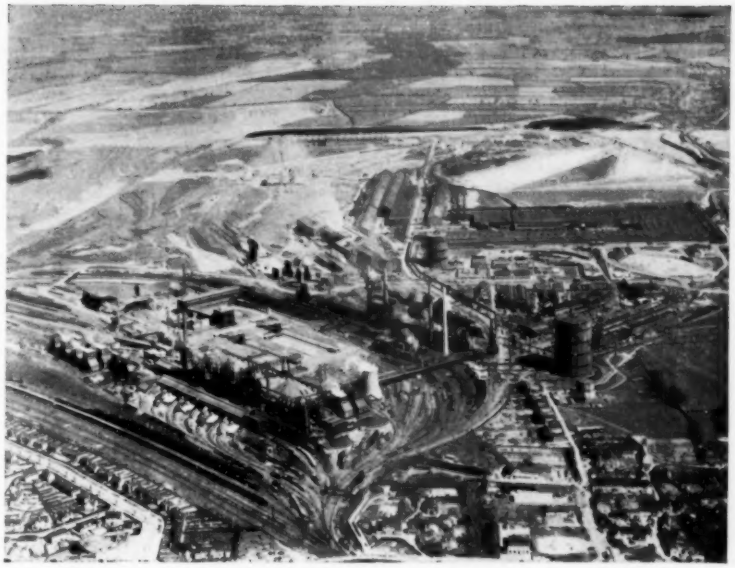
Midlands Area

The reconstruction scheme at ROUND OAK STEEL WORKS, LTD. provides for the installation of an entirely new melting-furnace plant comprising five 100-ton open hearth furnaces and an extension to the existing scrap bays. This construction, which is now almost completed, is being followed by the installation of a 37-in. cogging mill and the electrification of the present 28-in. and billet mills, which will be modified.

The new melting furnace plant, which comprises Venturi-type open-hearth furnaces, and the extensive modifications being effected will enable the ingot production to be increased to meet the rolling mill capacity and will promote more balanced operations leading to greatly improved efficiency. In place of the present 12- and 8-in. merchant mill a new cross-country mill is being installed, comprising an 18-in. cogging mill, seven stands of 16 in. 2-high mills, with two stands of 10-in. 2-high mills and a double-sided mechanical cooling bed is provided for dealing with bars from these mills.

Comprehensive steelmaking developments have been carried out by STEWARTS & LLOYDS, LTD. to ensure the success of their tube-making operations and the production of a wide range of materials in tubular form. The major part of the scheme at Corby dates from 1932 when there were three small blast furnaces producing Northants foundry iron; now, there are four modern blast furnaces with ore preparation plant, including three sinter plants, Bessemer, open-hearth and electric steelmaking units, rolling mills and tube plants.

The ore preparation plant has been in operation since 1948 and it has greatly improved blast-furnace operations. Northamptonshire ironstone is used exclusively and, since it is very variable in quality, an ore-mixing plant has been installed complete with crushing and screening units. The more recent extension is the new open-hearth melting shop which houses two 90-ton open-hearth fixed furnaces. These are designed for firing with oil fuel and coke-oven gas in varying proportions. Tube-making capacity has been extended by the addition of a fourth continuous weld mill capable of making tubes, from strip, up to 4½ in. diameter ; increasing it to 125,000 tons per annum.



An aerial view of the Corby Works of Stewarts & Lloyds Ltd.

Founded in 1865, to convert locally mined ores, the APPLEBY-FRODINGHAM STEEL CO., LTD. of the UNITED STEEL COMPANIES, LTD. has made continual progress, but probably the major developments were made between the wars when the new South Ironworks were planned and erected and put into operation in 1939. The plant in these works comprises crushing, screening, ore drying and ore bedding units, which prepare both local and Northamptonshire stone for the Frodingham, North Ironworks and South Ironworks blast furnaces. In addition to the ore preparation plant, a two strand Dwight-Lloyd sintering plant, and two blast furnaces, with 22-ft. diameter hearths and 66 Koppers coke ovens were installed. The developments at present in progress at these works include the doubling of the battery of coke ovens ; and doubling the ore drying and sintering plants. In the North Ironworks, after extensive experiments, No. 6 blast furnace was relined entirely with carbon blocks from hearth to stock. As this furnace was only put in operation in the spring of last year it is too early to comment on the results.

In 1946 work on the new South melting shop was started and put in operation in November, 1947. It contains two basic open-hearth furnaces of 300 tons capacity and a 600-ton active mixer. In the Frodingham section mills work on modification and electrification was also commenced in 1946, the first phase being completed in May, 1948, when the new 42-in. cogging mill commenced operation. Previous to this two blast-furnace gas-fired soaking furnaces, and a similarly heated ingot preheating furnace had been brought into operation. This project, which is concerned with the modernisation of the 32-in. section mill, has just been completed. The new mill will permit the use of 6-ton instead of 4-ton ingots.

The SCUNTHORPE WORKS of JOHN LYSAGHT, LTD. is being considerably expanded. The scheme involves an expenditure of £9½ million and excellent progress is being made. It provides for a new battery of 23 Woodall-Duckham coke ovens and additional coal crushing plant ; reorganisation of the handling of raw materials for the blast-furnace plant ; a blast furnace with a hearth diameter of 22½ ft., together with three stoves ; and a new Lodge-Cottrell electrostatic plant. These additions are expected to increase the output of pig iron to between 7,000 and 7,500 tons per week.

The capacity of the steel plant will be increased by the addition of a new open-hearth furnace and the enlargement of three furnaces, with a capacity of 65 tons, to 120 tons per furnace. Four new 175-ton electric overhead ladle cranes will be provided ; the scrap service to the furnaces is to be redesigned ; bogies, with roller bearings, carrying four pans of 30·6 cub. ft. capacity will service the steel plant ; the casting pit will also be served with roller-bearing bogies, each carrying 4 tons of ingot moulds ; and a new mould-handling shed will be provided.

The present rolling mills will be replaced by a new 42-in. reversing cogging mill which will serve a six-stand 19-in. Morgan continuous billet mill, with an electrically-operated continuous shear. A four-sided hot de-seaming machine will be introduced in the production line between the cogging mill and the first Morgan stand. Three Stein & Atkinson automatically-controlled recuparative soaking pits, having four cells to each pit, will be installed ; they will be heated by a mixture of coke-oven and blast-furnace gases. The mill, which at first will deal with about 10,500 tons of ingots per week, will have a capacity greatly exceeding this if necessary. It will produce blooms, billets, slabs, and sheet bar, and cooling banks will be installed to deal with the rolled material.

South Wales Area

Development work at the EBBW VALE WORKS of RICHARD THOMAS & BALDWINS, LTD. is designed to increase output and to improve the overall balance and efficiency of the operations and the quality of the products. These works are fully-integrated, comprising coke ovens, blast furnaces, Bessemer and open-hearth steel plants, slabbing mill, continuous hot-strip mill, three- and five-stand tandem cold-reduction mills, tinning lines, and ancillary departments.

Work is proceeding on 21 Woodall-Duckham Becker coke ovens additional to the 76 now operating. The two older 18-ft. single skip blast furnaces are being

converted to the standard design of Ebbw Vale "C" furnace, of 20-ft. diameter hearth, with double-skip hoist. Three new hot-blast stoves are being installed and additional ore bunkers completed; the electrostatic gas-clearing plant is being extended. An additional 120-ton basic open-hearth furnace, with the necessary extension of buildings, electricity and gas services is completed and a new department for the handling of ingots is in operation. The electrolytic tinning line, which has been in operation over two years, is fulfilling all expectations.

The various sections in the Group operated by this Company, at Redbourn, West Wales, Panteg, and the four works in the Midlands, are developed on sound and progressive lines. In the latter works, which have specialised in the production of silicon steel sheets for the electrical industry, and special coated and uncoated sheets for the automobile, gas, dairy and other industries, the hot- and cold-rolling facilities are being revised and augmented on the most modern lines. At the Panteg Works also, sheet-rolling facilities are being revised and augmented by the installation of modern cold-rolling mills and processing plant. These works specialise in the production of silicon, stainless, and other alloy steels, in billet, sheet bar, sheet and merchant bar forms.

What may ultimately be the greatest contribution to British industry is the immense project of the STEEL COMPANY OF WALES, which was formed by four of the largest firms engaged in the steel and tinplate industry. The main task of construction for this great project began in the spring of 1947. The project was to construct a completely integrated plant for the production of tinplate adopting the continuous strip mill method. The estimated cost was placed at £60 million.

The plan was to reconstruct and enlarge the blast furnaces, coke ovens and coal and ore handling plant at the Margam steelworks, in order to produce sufficient pig iron to meet the greater production of steel required, and to erect, adjacent to the Margam works, a new 80-in. continuous strip mill, together with a melting shop and ancillary plant, making one integrated works which will, when completed, extend for 4½ miles, and the estimated output of which will be 20,000 tons per week of steel strip and plate.

Originally, as an integral part of the scheme, there were to be two cold-reduction mills and a modern tinplate plant, the location of which were undecided. Since then it has been decided that one of these mills will be located alongside the continuous mill at Margam, the other, together with the tinplate plant, being located at Trostre, near Llanelly.

Reconstruction in the existing Margam steelworks is progressing steadily; the blast furnaces are being rebuilt for a weekly output of about 19,000 tons of pig iron; and a further 90 coke ovens are being added to the existing battery of 54. It is expected that the new works will be in full production next year.

Although it has been impossible to refer to all the developments made and in progress, sufficient has been said to indicate that the industry has made and is making revolutionary efforts to serve the many industries that depend on steel. No other industry has exceeded its production targets so consistently. It is very doubtful, therefore, whether a further experiment in public ownership, of such a complex industry, would maintain the rate of progress which has been achieved.

Acknowledgments

We are indebted to the many firms who have supplied information and illustrations for the preparation of the above outline of development in the industry.

Silver Jubilee Conference

A CONFERENCE to mark the twenty-fifth anniversary of the founding of the Electrodepositors' Technical Society will be held at the Grand Hotel, Eastbourne, from Wednesday, April 19th to Saturday, April 22nd, 1950. A specially attractive programme is being planned.

Technical sessions are being arranged at which papers will be presented and discussed on electropolishing, alloy deposition, and various other subjects connected with electroplating and protective metallic coatings. Further sessions will consist of general discussions at which well-known speakers will introduce topics of such practical interest as metal cleaning, electrical maintenance in the plating shop, and education in the plating industry. Technical sessions will occupy the morning and afternoon of the Thursday and Friday and the morning of Saturday, April 22nd. Considerable attention will be given to arranging attractive social functions which members and visitors and their ladies will be able to attend. Full particulars will be made available at a later date, but intending participants are advised to reserve the dates April 19th to 22nd, 1950.

Stellite

In the caption to Fig. 13, on page 132 of our last issue, reference is made to Stellite-faced, ordinary monel. As is well-known Stellite is a registered product of Beloro Smelting and Refining Co., Ltd.

Conference on Wire Ropes in Mines

A FOUR-DAY conference on Wire Ropes in Mines will be held between September 19th and 23rd., 1950, at Ashorne Hill, near Leamington Spa, Warwickshire. Arrangements for the conference are being made by the Institution of Mining and Metallurgy, in co-operation with the Institution of Mechanical Engineers, the Institution of Mining Engineers, the British Iron and Steel Research Association and other bodies.

The subjects to be dealt with include the design and manufacture of winding ropes, rope practice in the principal mining fields of the world, rope testing and failures, and Government regulations for the use of wire ropes in mines. Ropes for haulage, shaft guides and ropeways will also be discussed.

All those interested in problems connected with wire ropes used in mines are cordially invited to attend the conference, which will afford a unique opportunity to meet engineers from many parts of the world and to exchange views and experience.

A list of papers, with synopses, will be issued at an early date, and "preprints" of the papers will be published and circulated before September, 1950, so as to afford as much time as possible for discussion at the conference. It is hoped that, by giving ample notice of the date of the conference, arrangements can be made by all those who desire to attend. Further particulars can be obtained from the Secretary, Institution of Mining and Metallurgy, Salisbury House, London, E.C.2.

The Annealing of Nickel and High-nickel Alloys

In the last issue the importance of correct heating was emphasised in connection with the hot-working of nickel and the high-nickel alloys, Monel, "K" Monel and Inconel. The present article is concerned with the processing of these materials in which a sequence of heating and cooling operations is involved, generally referred to as annealing. These operations are discussed in detail.

ANNEALING is a process involving a sequence of heating and cooling operations and usually implying relatively slow cooling. Applied to metals and alloys, it is a process of heating for a definite time at a predetermined temperature, followed by cooling to produce a change in mechanical properties. This change is usually a complete or partial softening as a result of recrystallisation, but sometimes there is slight hardening as a result of equalisation of stress in the material. For some materials, such as the steels, cooling must be slow; for others, such as nickel, high-nickel alloys and copper, it may be either rapid or slow; and in a few special cases, such as the precipitation-hardening alloys, including "K" Monel, it must be rapid.

SOFT ANNEALING

Metals and alloys which have become hardened by cold-working operations require softening before further cold-working. The thermal treatment which will produce this effect is known as *annealing* or *soft annealing*.

The most important factors to be observed in soft annealing of the high-nickel materials are :—

Protection against embrittlement and excessive oxidation by providing non-scaling atmospheres with low sulphur content. Furnaces must be designed so that the combustion reactions are complete before the hot gases strike the metal. Control of the temperature and of the time at temperature to avoid excessive grain growth.

Coarse- or large-grained material is unsuitable for most cold-working operations. Maximum workability is obtained with material annealed to remove the effect of previous cold-working without allowing appreciable grain growth to occur. This condition gives the best combination of ductility to permit extensive deformation, strength to withstand the action of the tools, and surface quality to facilitate polishing and finishing.

Injury to the surface is avoided by using fuel of low sulphur content; having heating atmospheres free from active oxygen; cleaning thoroughly to remove drawing compounds, paint marks and any other adherent substances which might contain sulphur, lead or other harmful ingredients; supporting the work to avoid contact with scale, slag and cinder on the hearth of the furnace.

Open Annealing

This is the method most widely used for bright annealing. While hot, the metal or alloy to be softened is protected from oxidation in a fuel-fired furnace by the gases of combustion and in an electric furnace by the introduction of special atmospheres referred to later. The furnace should be at the selected, uniform temperature before the work is charged. During preliminary heating of a fuel-fired furnace the burners may be adjusted for optimum combustion but the air supply should be reduced before charging the work to give the excess of reducing gases required for clean heating. Also, the dampers should be wholly or partially closed to give a positive gas pressure.

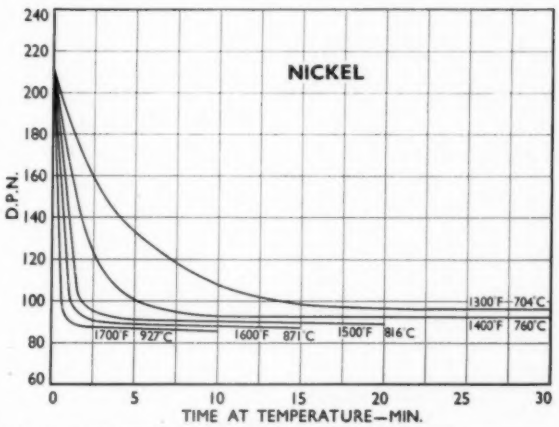


Fig. 1.—Approximate time required at various temperatures to produce different degrees of softness in nickel by open annealing.

Figs. 1–7 show the average times for which nickel, Monel, "K" Monel and Inconel must be held at the indicated temperatures to produce the required degree of softness and grain size. The curves apply to single pieces of material in the furnace, and if pieces are stacked

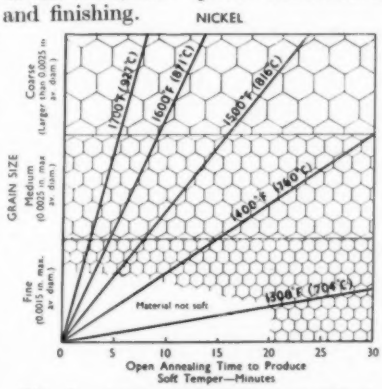


Fig. 2.—Approximate time required at various temperatures to produce different grain sizes in nickel by open annealing.

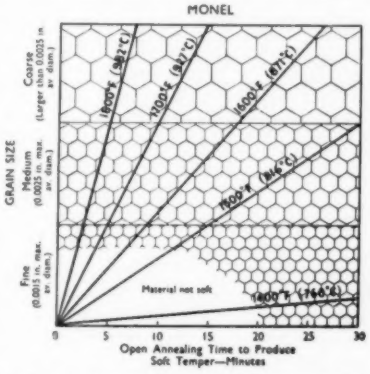


Fig. 4.—Approximate time required at various temperatures to produce different grain sizes in Monel by open annealing.

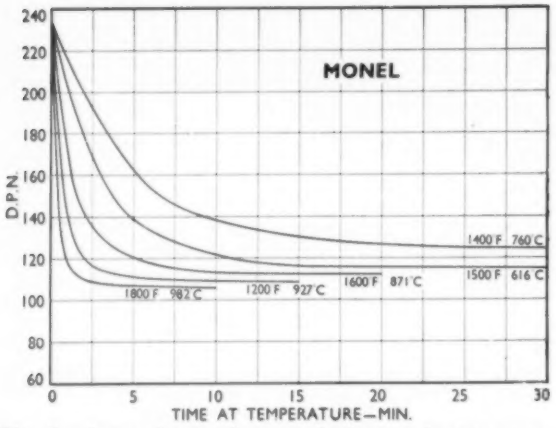


Fig. 3.—Approximate time required at various temperatures to produce different degrees of softness in Monel by open annealing.

or are shadowed by other pieces, this must be considered in determining both the time the work should remain in the furnace and the annealing temperature in order to avoid a coarse-grained structure.

For example, a single piece of hard Monel sheet must be annealed to a hardness of 115 D P N. to be suitable for further spinning operations. The furnace temperature available is 1,600° F. (870° C.). The 1,600° F. curve in Fig. 3 shows that this degree of hardness can be obtained in 4 minutes at temperature. Fig. 4 further shows that the time must not be allowed to exceed 9 minutes for a fine grain, or 17 minutes for a medium grain.

As a second example, hard Inconel strip is to be annealed to 160 D.P.N. for further cold-working. With a furnace temperature of 1,800° F. (980° C.), Fig. 6 shows that this softness can be obtained in 4 minutes. Fig. 7 further shows that the time must not exceed 16 minutes for a fine grain. Similar procedures should be followed for nickel and other nickel alloys.

Some classes of work, particularly pieces for spinning, require softer annealing than others. The more severe the nature of the subsequent cold-working operation, the longer must be the time at temperature, without, however, going beyond the limits for grain size recommended later in this article. The time/temperature ranges given in Table I are sufficiently broad to provide a satisfactory latitude in handling the work in and out of the furnace. The shorter periods do not yield full softness but will condition the metal for cupping, drawing and stamping.

Continuous belt-conveyor furnaces are widely applied for annealing of pressings, blanks, sheets and light coils of wire and strip. As they have more or less open ends, these furnaces consume a relatively large volume of atmosphere gas, and the cheaper atmospheres derived from coal gas are therefore preferable whenever suitable.

Closed (Box or Pot) Annealing

Because of the lower temperatures used for box, or pot, annealing, more time is required. Temperature control is therefore not so critical, nor are the type of furnace and fuel used so important as in open annealing, because the work is protected from contact with the furnace atmosphere. Box annealing necessitates heating the container to a slightly higher temperature than that required for the work. In most cases, the weight of the

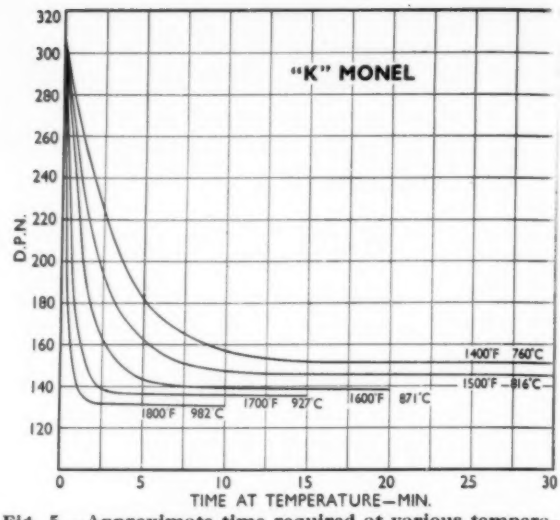


Fig. 5.—Approximate time required at various temperatures to produce different degrees of softness in "K" Monel by open annealing.

container exceeds that of the work, which increases the amount of fuel required, the heating time and therefore the cost. Consequently, box annealing is used much less generally than open annealing. Such articles as small pressed parts, rivets and similar work are, however, preferably annealed in boxes because of greater ease in handling.

The method is not suitable for softening "K" Monel, which must be cooled rapidly to prevent age-hardening.

TABLE I.—ANNEALING TEMPERATURES FOR DRAWN SHAPES AND SPINNINGS OF MONEL, NICKEL AND INCONEL

Material	Temperature °C.	Temperature °F.	Time at Temp. Minutes
Nickel	815	1500	3– 6
	925	1700	1– 4
Monel	870	1600	5–10
	980	1800	1– 4
Inconel	980	1800	7–15
	1065	1950	2– 5

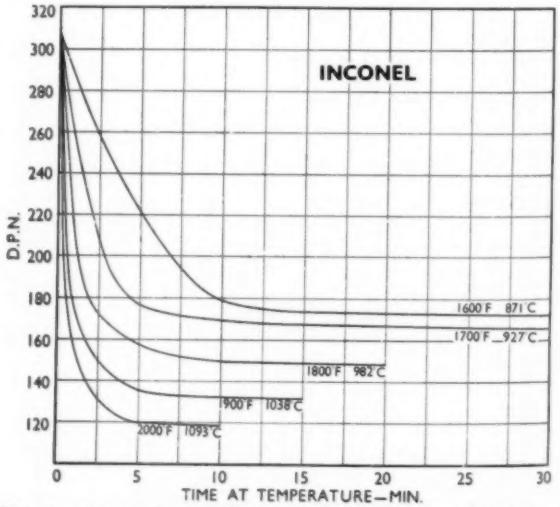


Fig. 6.—Approximate time required at various temperatures to produce different degrees of softness in Inconel by open annealing.*

* It is recomended that a minimum temperature of 980° C. (1,800° F) be used.

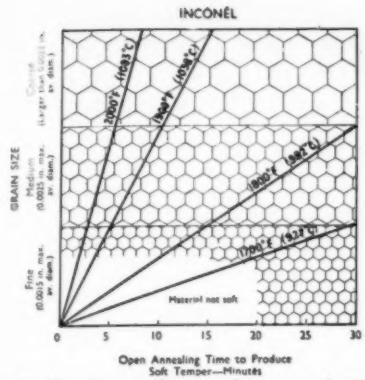


Fig. 7.—Approximate time required at various temperatures to produce different grain sizes in Inconel.

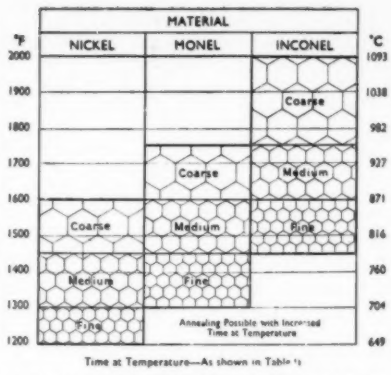


Fig. 8.—The relation of grain size to temperature in long time closed annealing.

Figs. 8 and 9 show the average time for which nickel, Monel and Inconel must be held at the temperatures indicated to produce the specified grain size and softness. For example, Inconel can be soft annealed to have a fine grain size at temperatures up to 870° C. (1,600° F.). Nickel, on the other hand, will be soft, although coarse-grained, if annealed about 790° C. (1,450° F.), while Monel may be soft annealed at temperatures below 870° C. (1,600° F.) without producing a coarse grain.

Except for Inconel, bright annealing in boxes and pots is secured by introducing an appropriate sulphur-free, dry reducing gas into the container during the cooling part of the cycle. Inconel cannot be bright annealed in pots even in hydrogen, because of the practical difficulty in maintaining the hydrogen atmosphere at the high degree of purity required to reduce the chromium oxide.

Annealing boxes may be made of mild steel, cast iron or preferably of a heat-resisting alloy such as nickel-chromium-iron. A wide variety of shapes and sizes can

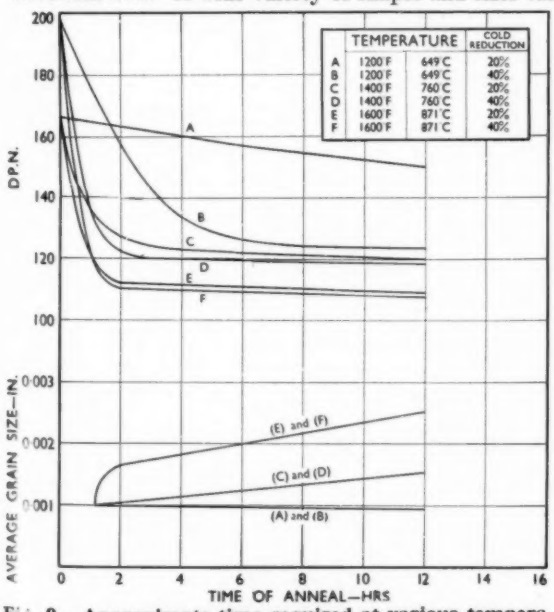
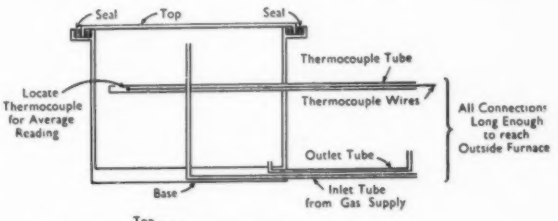


Fig. 9.—Approximate time required at various temperatures to produce different grain sizes and degrees of softness by closed annealing.

be used. One type is shown in Fig. 10. Welded steel boxes of this type are usually cylindrical in shape, their height being equal to or slightly greater than their diameter. To secure more uniform heating, boxes should, wherever possible, be designed to have a centre flue equivalent to half the diameter of the box so that when the box is raised on rails a good circulation of hot gases is obtained. Another good type of box is shown in Fig. 11 and consists of a flat, flanged bottom on which the work is stacked, and an inverted bell or cover. This is often described as a "top-hat cover."

The box should be large enough to allow for loose packing of the parts but with little excess space. A

Fig. 10.—Pot-type annealing box with connections.



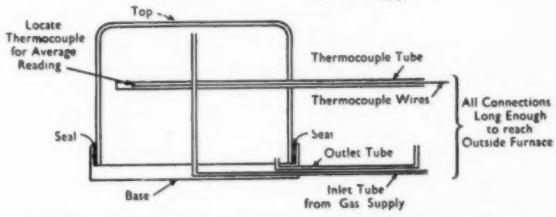


Fig. 11.—Bell-type annealing box with connections.

suitable thermocouple should be located about the centre of the charge, usually by running the connection through a gas-tight joint. After the work is charged, the box should be luted with fire clay or sealed with sand or other refractory material. A small quantity of charcoal should be packed in the box at points where air could enter. It is very important that the charcoal be preheated at 650°–815° C. (1,200°–1,500° F.) for at least 3 hours to remove any occluded oxidising gases. The box should be provided with a hole to admit a small pipe for introducing the dry, reducing gas immediately the box is withdrawn from the furnace, or on shutting off the burners. Hydrogen, town's gas, cracked ammonia or mixed reducing gases free from sulphur may be used. The flow should be regulated to maintain a positive

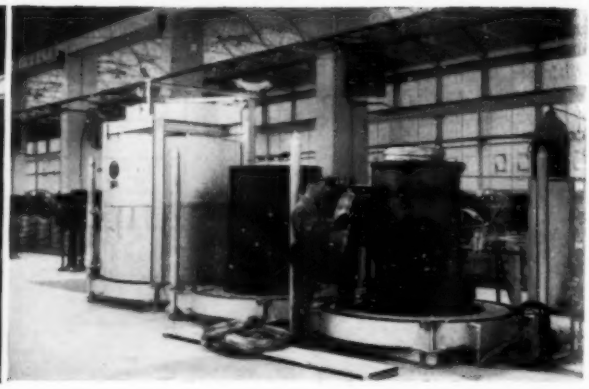
TABLE II.—TEMPERATURE AND TIME FOR CLOSED ANNEALING OF NICKEL AND NICKEL ALLOYS

Material	Temperature °C.	Temperature °F.	Time at Temp. Hours
Nickel	705	1300	3
	760	1400	1
	815	1500	½
Monel and "K" Monel*	760	1400	3
	815	1500	1
Inconel	870	1600	3

* Quench or cool in air, when possible.

Courtesy of Birlec Ltd.

Fig. 12.—Two belt-conveyor furnaces used for annealing Monel sheet and strip.



Courtesy of Birlec Ltd.

Fig. 13.—Three-hearth bell-type furnace suitable for batch annealing of nickel-alloy wire.

pressure within the box so that the gas, if combustible, will burn gently at all outlets.

The time required in the furnace depends on the weight of the box, the amount of material in the box and the heating capacity of the furnace. Wide variations in these factors make it difficult to estimate the total time of heating, but Table II gives the required time at temperature, to which must be added the time for heating up.

An alternative method of closed annealing is by direct heating of the parts in the furnace proper. It is necessary that the furnace be checked carefully for air leaks and that good control of the furnace atmosphere be provided. Since it is difficult to obtain good results from direct gas heating, it is suggested that the method be applied only in electric or radiant tube furnaces. Direct heating is usually not capable of producing bright annealing, especially with Inconel and " K " Monel.

For batch annealing for rod, wire, strip, tubing, etc., electric or gas-fired furnaces of either bell or pit type are standard equipment. In the bell type, the charge rests on a stool on the hearth of the furnace and is covered by a light sheet-metal cover which engages with a powder or liquid seal around the hearth to form a gas-tight enclosure. The furnace proper is in the form of a bell which is lowered over the covered charge. Three or more hearths are provided with each furnace. (See Fig. 13).

After the hearth is loaded, its cover is put into position and a supply of protective atmosphere is fed into it until all air has been purged out. The furnace, already at temperature, is lowered over it and kept in position until the charge is fully heated. At the end of the required annealing time, the furnace is removed, the atmosphere outlet from the hearth closed and the charge is allowed to cool under a slight pressure of atmosphere gas.

In the pit-type furnace, the heating chamber is sunk below the floor and the charge, in a purged and sealed container, is lowered into it. Operating procedure is similar to that described for bell furnaces.

Salt Bath Annealing

This method is used for special work with small parts. Inorganic salts, such as chlorides and carbonates of sodium and potassium, which are relatively stable at temperatures considerably above their respective melting points, are melted in large containers. Steel pots are satisfactory for use at temperatures up to 700° C. (1,300° F.) but at higher temperatures, heat-resisting nickel-chromium-iron alloy pots can be used. For neutral salt baths operating at 750°–900° C. and heated electrically by immersed electrodes, ceramic containers are preferred.

When nickel or high-nickel alloys are to be annealed in fused salts, particular care must be taken to prevent embrittlement by removing all traces of sulphur before immersing the work. This can be done in 2–3 hours by adding a small amount of a 3 to 1 mixture (by volume) of powdered borax and powdered charcoal to the fused salts. If small test pieces of nickel or Monel are not embrittled after 3–4 hours in the purified salt bath, the desulphurising treatment has been sufficient.

The material to be annealed is placed in the molten salt and rapidly absorbs heat. After being annealed, the work should be quenched in water to free it from particles of the salt mixture. The annealed material will not be bright and must be flash pickled. Table III gives some useful salt mixtures.

TABLE III.—MELTING POINTS OF SALT MIXTURES

	Mixture		Melting Point		Working Range	
	Ingredient	Per cent. by weight	° C.	° F.	° C.	° F.
1	Potassium Chloride	56	660	1220	700–815	1300–1500
	Sodium Chloride ..	44				
2	Sodium Chloride ..	72·6	675	1247	730–815	1350–1500
	Sodium Fluoride ..	27·4				
3	Barium Fluoride ..	70	790	1454	845–925	1550–1700
	Calcium Fluoride ..	15				
	Magnesium Fluoride	15				
4	Barium Chloride ..	83	844	1551	900–1000	1650–1850
	Barium Fluoride ..	17				

Electric Resistance Annealing

This also is a special method. An electric current is passed through the material to be annealed which acts as a resistor and is heated up rapidly. The method has seldom been satisfactory in commercial work. Temperature control is difficult unless the material is uniform in size. Furthermore, it is impossible to provide a suitable reducing atmosphere for bright annealing. Results are generally irregular.

Torch Annealing

If the annealing furnace is too small to hold the work, sections of large equipment which have become

locally hardened by the fabricating operations can be annealed with the flames of oil or acetylene torches, adjusted to be highly reducing. The method is not a good one for general use because it gives irregular and insufficient annealing with heavily oxidised surfaces. If it is the only method available the following procedure should be adopted.

Heating must be gradual. It may be facilitated by temporary brick construction to localise the heat and to permit higher temperatures to be attained. The work should first be gently warmed with sweeping motions of the torch and should not be brought to the annealing temperature until it is preheated sufficiently to prevent cracking due to sudden release of stresses.

DEAD-SOFT ANNEALING

When the high-nickel materials are annealed at higher temperatures and for longer periods than those prescribed for soft-annealing, a condition commonly described as " dead soft " is obtained. In this case, the hardness will range 10–20% lower than the figures for the " soft " condition. This degree of softness cannot be obtained without increasing the grain size of the material. This process should, therefore, be used only for those few purposes for which grain size is immaterial.

Articles to be cold-worked subsequently in such operations as drawing and spinning should never be annealed dead soft, since this will produce a coarse grain. Heavy plate sections and thick-walled tubing that are required only to negotiate certain bending operations may, however, be annealed dead soft to facilitate working because the coarse grain produced does not affect the material adversely.

TEMPER OR PARTIAL ANNEALING

The process of heating a hardened metal or alloy at such a temperature and for a period sufficient to accomplish only partial softening is known as " temper annealing," " semi-annealing " or " partial annealing." In practice, it is limited to light sections and is usually applied only to strip and wire, but is valuable for operations where fully annealed material is not required. Any of the previously mentioned methods of annealing may be used, although the continuous method is best suited for the purpose. Careful regulation of time at temperature is essential. Data given in Table IV at 400°–600° C. (752°–1,112° F.) illustrate the results which can be obtained.

LOW TEMPERATURE (STRESS EQUALISING) ANNEALING

Both cold-worked and hot-worked nickel and nickel alloys require a low-temperature thermal treatment to develop their optimum combinations of strength and ductility, and to guard against instability of dimensions upon subsequent machining. This treatment is often erroneously called stress-relief annealing or normalising, whereas it neither relieves nor removes all stresses nor does it alter the grain structure. It equalises, or homogenises, the material by dissipation of local concentrations of stress, so as to render it satisfactory for precise engineering applications.

The proper low-temperature or stress-equalising, thermal treatment for this purpose depends upon the nature of the preliminary fabrication or working. Hot-worked material requires a higher homogenising temperature than cold-worked material. The thermal treatment produces a considerable increase in mechanical properties, especially the proportional and elastic limits and proof stress, without change in elongation and reduction of area.

Figures in Table IV show the effects of soft, stress-equalizing and temper-annealing on the mechanical properties of cold-drawn Monel. It will be seen that above 350° C. strength and hardness decrease fairly rapidly and ductility increases. Similar results are obtained with nickel, but Inconel can be stress-equalised up to 425° C., without decrease in strength. With " K " Monel, although slight age-hardening results after heating at these low temperatures, this treatment is unnecessary after age-hardening.

TABLE IV.—EFFECT OF ANNEALING TEMPERATURE UPON MECHANICAL PROPERTIES AT ROOM TEMPERATURE OF A 1½ IN. DIAMETER COLD-DRAWN MONEL ROD

3 hr. at Temperature	Proportional Limit tons/sq. in.	0·5% Proof Stress tons/sq. in.	Maximum Stress tons/sq. in.	Elongation % on 2 in.	Hardness D.P.N.
None—rod as cold-drawn	20·1	44·4	48·4	20·5	220
200° C. (392° F.) ..	27·9	46·0	49·6	20·0	240
250° C. (482° F.) ..	30·1	46·5	50·5	20·6	240
300° C. (572° F.) ..	31·2	45·1	50·2	20·8	230
350° C. (662° F.) ..	29·7	43·7	49·8	22·2	220
400° C. (752° F.) ..	26·8	40·7	49·1	25·2	210
500° C. (932° F.) ..	25·9	36·2	46·2	27·0	205
600° C. (1112° F.) ..	23·4	32·6	44·3	30·8	185
700° C. (1292° F.) ..	10·0	15·2	37·8	44·2	120
750° C. (1382° F.) ..	8·9	15·1	37·3	43·5	115

Specimens heated for 3 hours and air-cooled. Stress-equalising annealing, 200°–300° C.; temper annealing, 400°–600° C.; soft annealing, 750° C.

Cold-worked Material

Recommended stress-equalising temperatures for cold-worked material are :—

Monel and nickel, 275° C. (525° F); Inconel, 425° C. (800° F.).

Long exposures at this temperature are not harmful, as shown in Table V, which also confirms the necessity for the thermal treatment in order to produce the maximum proportional limit.

TABLE V.—EFFECT OF STRESS-EQUALISING ANNEALING AT 275° C. (525° F.) UPON THE PROPORTIONAL LIMIT OF A 1 IN. DIA. COLD-DRAWN MONEL ROD (20% REDUCTION)

Time at Temperature hours	Proportional Limit tons/sq. in.
As drawn	17·9
1	36·6
2	36·6
4	36·2
8	35·7
16	35·7

Hot-worked Material

Table VI summarises data, indicating that the optimum time and temperature for stress-equalising

TABLE VI.—EFFECT OF TEMPERATURE UPON THE MECHANICAL PROPERTIES AT ROOM TEMPERATURE OF ⅞ IN. HOT-ROLLED MONEL PLATE

3 hr. at Temperature		Proportional Limit	0·2% Proof Stress	Maximum Stress	Elongation on 2 in.	Reduction of Area	Hardness
° C.	° F.	tons/sq. in.	tons/sq. in.	tons/sq. in.	%	%	D.P.N.
None	None	16·1	27·0	38·2	32·9	67·0	170
300	572	21·0	27·2	38·4	33·6	65·4	165
500	932	21·4	26·8	38·4	35·0	67·0	165
550	1022	21·4	26·1	38·5	34·0	67·1	160
575	1067	22·3	25·7	38·6	35·0	65·7	155
600	1112	22·1	25·6	38·4	35·4	65·3	158
650	1202	20·5	24·5	37·9	35·7	65·0	153
800	1472	12·1	14·3	35·7	44·3	68·3	120

hot-worked Monel is 3 hours at 550°–600° C. (1,022°–1,112° F.).

Springs

Monel, "K" Monel and Inconel springs are made from heavily cold-drawn wire or cold-rolled strip and should be stress-equalised after coiling. Preferred treatments are :—

	°C.	°F.	Time
Monel and "K" Monel	340	650	1 hr.*
Inconel	480	900	1 hr.

* Not necessary with "K" Monel springs which have to be finally age hardened.

(To be continued)

Staff Changes and Appointments

MR. I. A. R. STEDEFORD, Chairman and Managing Director of Tube Investments, Ltd., left Britain by the s.s. *Queen Elizabeth* on February 15th on a business tour, during which he will visit the United States, Australia, New Zealand and South Africa. He will be accompanied by MR. E. D. E. ANDREWES, Managing Director of the Tube Investments export organisation TI (Export), Ltd. and will return to England in mid-April.

DR. J. PEARSON has been appointed Head of the Chemistry Department of the British Iron and Steel Research Association in succession to Dr. F. D. Richardson. He has been Head of the Chemistry Section of the BISRA Laboratories at Sketty Hall, Swansea since 1946, where he has worked on the disposal of spent pickle liquor, gas pickling of steel, preparation of replicas for surface finish assessment, etc.

Dr. Pearson is a graduate of London University and has previously worked with Messrs. Tube Investments, Ltd., and with the Armament Research Department of the Ministry of Supply at Woolwich. He received the degree of Ph.D. in 1946 for research on the polarography of nitrocompounds. In his new post, he will be responsible for research on physical chemistry of iron and steel-making, refractories and problems of corrosion.

DR. FRANKLIN T. PETERS has been assigned to the newly-formed Development Department of Glyco Products Company, Brooklyn, N.Y. He has been with the company, in the Sales Department, since 1947 following ten years with E. I. du Pont de Nemours & Co. in research positions.

MR. D. W. HAMMOND has been appointed Works Director and MR. C. WHITAKER Production Manager of William Jessop & Sons, Ltd., Sheffield.

MR. C. R. DUNN, late European Representative of British Timkin, Ltd., has been appointed Sales Manager, Fischer Bearings Co., Ltd., Wolverhampton, a subsidiary of the British Timken company.

MR. RICHARD MILES, Managing Director, has been elected Chairman of Head, Wrightson & Co., Ltd. in succession to the late Sir Guy Wrightson, Bt. SIR JOHN WRIGHTSON, Bt. has been appointed Vice-Chairman.

MR. H. A. A. WHILE, M.B.E., has been appointed London Manager of the United Steel Companies, Ltd. Mr. While will remain in charge of the Railways Department.

MR. V. A. LOWINGER, C.B.E., retired from the Chairmanship of the International Tin Research and Development Council, which controls the Tin Research Institute, on December 31st last, as he is no longer permanently resident in this country. His successor is Mr. G. F. A. Burgess, a Joint Managing Director of the British Metal Corporation, Ltd.

MR. F. R. WISKAR, Steel-plant Manager at Dorman Long's Britannia Works has been appointed to a similar post at the Company's Cleveland Works.

MR. CECIL HIPWELL, a Director and General Manager of Redpath Brown, one of the structural engineering subsidiaries of Dorman, Long & Co., Ltd. has been appointed a Special Director of the parent company.

MR. L. V. AMOR, Chief Engineer of Guest, Keen, Baldwin's Iron and Steel has retired. Mr. Trevor Jenkins is now acting chief engineer.

MRS. C. F. TIPPER has been appointed a Reader in the Engineering Department of the University of Cambridge.

MR. W. G. CALDER has been appointed a Director of Keith Blackman, Ltd., in place of MR. C. TUSON, who resigned at the end of last month.

Mr. A. Glynne Lobley

THE announcement of the death of Mr. A. Glynne Lobley, M.Sc., A.M.I.E.E., on January 16th would come as a great shock to his colleagues and to his many friends. Born at Hanley, Stoke-on-Trent, he was educated at Newcastle-under-Lyme High School and the Victoria University Manchester. For some years he lectured at Manchester and Sheffield Universities on electrometallurgy and later founded Birmingham Electric Furnaces, Ltd., now well known as Birlec, Ltd., furnace manufacfacturers of world-wide reputation, of which Mr. Lobley was Managing Director at the time of his death. In addition to his business activities Mr. Lobley took an active part in political affairs ; he represented Gravelly Hill Ward on the City Council from 1939 to 1945 and was Chairman of the Technical Schools Sub-Committee. At the time of his death Mr. Lobley was Hon. Treasurer of the Erdington Ward Unionist Association, Bailiff of Sir Josiah Mason's Orphanage, Hon. Associate of Central Technical College, Birmingham, Member of the Institute of Fuel, Birmingham Rotary Club, and Masonic Lodges, Loyal Service and St. Trillo. He will be greatly missed by a very wide circle of friends in many fields of activity.

The Aluminium Industry—To-day and To-morrow

By Col. W. C. Devereux, C.B E.

Managing Director, Almin Limited

The aluminium industry has become one of the basic industries and, in view of the fact that commercial production is practically confined to the present century, this can be taken as an indication of its remarkable progress. It is apparent that the advantages of lightness, strength and durability of aluminium alloys combine to offer great economic advantages in use. Although the industry has not yet reached stable maturity, the author is of the opinion that, with continued technical developments and the application of research, vast opportunities for the industry lie ahead in an increasing variety of services.

ALTHOUGH the final figures are not yet available the aluminium alloy industry of 1949 looks, statistically, very like that of 1948. Consumption of virgin and secondary aluminium and output of semi-manufactures are about the same in total and in proportion as in 1948. As a matter of fact, the total output is not very different from that of 1947.

What of the future of the aluminium industry in the decade ahead? It will be fashioned largely, of course, by the general political, economic and industrial events. It is one of the penalities of a basic industry that as it grows to importance so its affairs become more closely linked with the general industrial trends. When the aluminium industry was very young, before the war, it could, and did, continue a rapid expansion through depressions and slumps because it was so small and inexperienced and a vast expanse of virgin markets stretched out before it. To-day it is a great mass-production industry with great resources of production and scientific knowledge; we know a great deal more about the uses and behaviour of aluminium and we can now tackle great projects with confidence in our technical ability and resources, but the great plants we have built need big orders to keep them producing smoothly.

In the past four years we have learnt some valuable lessons and acquired a great deal of knowledge, but we still have much to learn about the advantages of aluminium in service. In 1949 we have learned again, what some of us tended to forget in the palmy days of the seller's market, that aluminium finds its most ready application where all its advantages of lightness, strength and durability combine to offer the greatest economic advantages from its use. In other words we are having to sell aluminium in competition with other materials instead of offering it as a readily available substitute for traditional materials in short supply.

Ironically it is we who are now haunted with uncertainty about the future of raw material supplies. It is inconceivable that means will not continue to be found to assure their delivery in the future, but there is still in our minds the uncertainty, notwithstanding the overriding world capacity for the production of aluminium. We are told so little of the details affecting the supply of aluminium from Canada or of the progress of projects within the sterling area that we cannot forget this threat for very long.

If we had only to consider the efficiency and strength of the fabricating industry in this country, and its technical ability, we could say with confidence that we could meet all forseeable demands for aluminium products and meet competition from any quarter. We can, and do, make the most of our opportunities and fight to maintain the markets we have won, but we can only manoeuvre within the narrow limits set by the national economy and world events.

To look very far ahead in an effort to predict the future of the United Kingdom aluminium industry, against such a background would, I fear, be unwise. The volume of primary aluminium available will depend on the dollar situation for several years to come. Prices will be influenced by the dollar exchange rate and markets will be influenced by prices relative to other materials.

Not only is Canada the only available source at present for the quantity of aluminium required to keep the United Kingdom fabricating industry operating at its full planned capacity, or even at its present level, but is also the lowest cost producer and, with the possible exception of Norway, it is likely to remain so for many years to come.

Although several schemes for large scale production within the sterling area are being developed (very little information is available about their progress) it will be some years before they can make any real contribution to the problem. On the other hand, several European countries are rapidly increasing their capacity and some already have an exportable surplus. Consequently there is considerable pressure on U.K. with its large fabricating industry to purchase European aluminium and so help European economy and save E.C.A. dollars. Generally, however, European prices are higher than Canadian and in any case it would still be necessary to obtain the balance of aluminium which Europe could not supply and if Canadian production were reduced their costs would tend to rise so that the net result would be a further increase in the cost of aluminium which in turn would tend to reduce consumption. In fact we might be confronted with a spiral of decreasing production and increasing prices.

For strategic and short-term economic reasons also, there is a general tendency throughout the world for self-sufficiency in aluminium. Many of the schemes now being put in hand for aluminium production and fabrication are bound to be uneconomic either by reason of their relatively small capacity or because of the lack of cheap hydro-electric power or other natural resources and of sufficiently large commercial markets.

This then is the dilemma: Canada offers low cost aluminium in sufficient quantities to meet all our immediate requirements and indeed is largely dependant upon the demands of the U.K. fabricating industry to keep its great producing plants working economically. Canadian aluminium, however, has to be paid for in hard currency so there is considerable pressure to seek supplies from other sources.

Thus, the industry, and the Government departments concerned with it, face formidable problems which will call for very careful handling and patient negotiations by both parties if our leadership in this field is to be maintained, and we are to weather the exchange difficulties of the next year or two without jeopardising the great opportunities which are already on the horizon.

Technical Development

The technical outlook is a lot brighter. We know a great deal more about the advantages and worth-while uses of aluminium than we did five years ago.

To offset the advances in raw materials and labour costs we are making great strides in technical efficiency and productivity. New plant now being laid down will still further improve our productive efficiency in rolling, extruding and die casting. The careful study of metal economy and technical advances in extrusion, die casting and other branches is giving the user the opportunity of increased efficiency in design to reduce the cost of metal in his products without loss of strength or durability. Improved methods of forming and joining are reducing costs of finishing. Looking a bit further ahead, new processes of surface finishing such as electro-polishing, electro-plating, vitreous enamelling and others bid fair to increase the range of uses and reduce the cost of finishing aluminium products. Welding research and development has taken a new lease of life from recent technical improvements and bids fair to reduce the cost of assembling aluminium structures, but it may be some time before this work is finalised.

Developments in new alloys, a field which has seen little activity since the war, are looking up again and we may shortly see developments in new alloys specially adapted for weldability, surface finishing, bearing properties and for more efficient fabrication.

Market Development

New uses for aluminium are being found at an ever increasing rate, due to the general acceptance of aluminium by engineers and designers and our ability, based on experience, to make more definite statements as to its technical suitability far more often than in the past.

Usually the deciding factor is price compared with alternative material. For many uses aluminium is preferred and will command a higher price, but the margin is limited.

It is encouraging to find, now that shortages of traditional materials are easing off, how few applications which really utilise the qualities of aluminium to advantage, revert to other materials, and it is very gratifying to see how soundly, in the main, we have chosen our markets.

Standing on the threshold of a new decade we wonder what it has in store. In the decade just behind us a revolution has taken place in the aluminium industry. We cannot claim that this revolution has been entirely of our own making—it has been the product, mainly, of far greater events. So in the next decade our development will be tied to the rise or fall of human fortunes, but I believe that we shall go on developing—inspired by our successes and learning from our failures as we have always done. At this point in our history we have just emerged from comparative obscurity to a general acknowledgment of our place in the industrial society.

Although I write with some reserve about prospects of expansion in the next few years, I do believe, indeed I think it inevitable, that as a long term trend the aluminium industry is expanding rapidly and will continue to do so for many years to come. We in the industry who have taken part in its remarkably rapid growth are apt to feel at times that it has already reached maturity, but although it has become an important basic industry it still has a very long way to go.

It is as well to remind ourselves that commercial aluminium production is practically confined to the 20th century. That more aluminium was produced in 1943 than was produced in the world before 1939. That all the aluminium produced so far is about equal in weight to the amount of steel produced in 1949 in U.K. alone.

Add to this the fact that every week brings new uses for aluminium and new experiences of its behaviour in an increasing variety of services. No, there is far too much momentum, far too many unexplored uses and markets, and far too much still to learn of its properties for the production and use of aluminium even to indicate when the industry will achieve a stable maturity.

In conclusion I see ahead vast opportunities for the aluminium industry. With world economy going from crisis to crisis we shall undoubtedly have many worries and set-backs in the years immediately ahead but overall the industry must expand. Firstly, industrial production is increasing rapidly and existing markets for aluminium will expand with it. Secondly, there are still far more uses for aluminium to be developed than have been discovered to date. Thirdly, this industry is still in the forefront of scientific research and many of the post-war projects now at last approaching completion are beginning to yield important new knowledge which will increase the rate of development.

Aluminium School Buildings

ALTHOUGH orders for aluminium school buildings continue to come in from all parts of Britain, increasing interest is being shown by overseas authorities. In this connection one manufacturer—The Bristol Aeroplane Company (Housing), Ltd.—in a survey of the position in mid-November showed that 70 school buildings had been completed, 31 were in course of construction, while another 60 were scheduled for erection in the near future—a total of 161 orders in all—an increase of 40 orders in the four months since a previous survey was made. Three prototype schools ordered by the Government of Victoria, Australia, are being erected. Interested inquiries have been received from authorities in the Argentine, Mexico and various European countries.

Meanwhile structural development continues; the latest improvement to be introduced is a new component, a load-bearing internal partition. This stressed panel dispenses with the use of internal and external buttressing in rooms up to 28 ft. in length. The resultant saving in aluminium has enabled prices to be maintained, despite the rise in cost of materials due to devaluation of sterling.

Progress of the Aluminium Industry

Post-war Production Activities

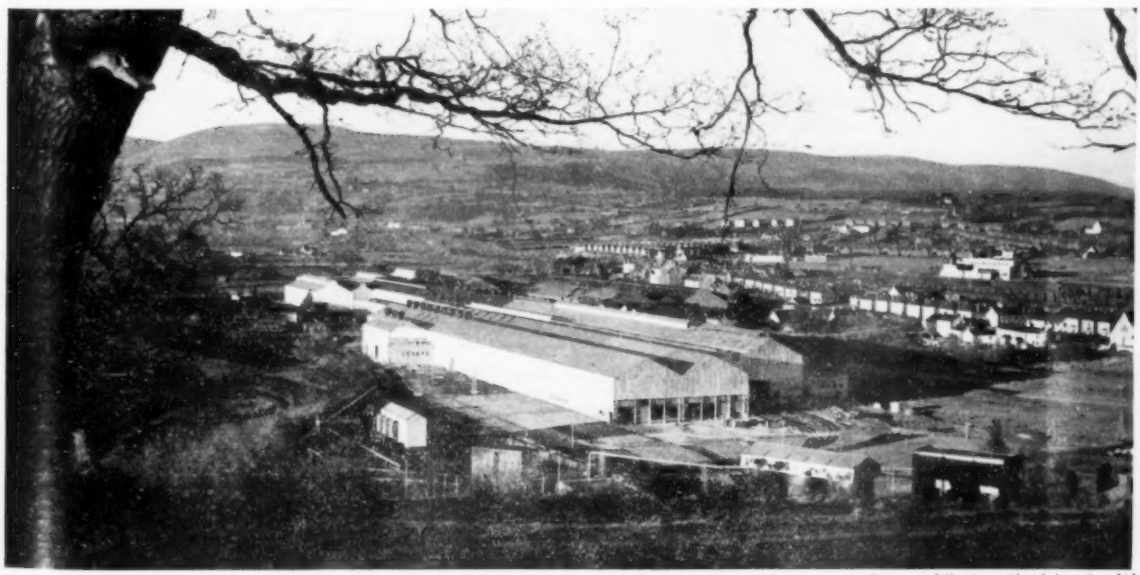
War conditions caused development in the industry to proceed at such a rapid rate that it far exceeded pre-war requirements, and great extensions, in buildings and plant, were made to meet abnormal demands. The change-over from the production of aircraft products to meet post-war needs was therefore complicated and necessitated many adjustments and alterations in utilisation and sometimes in the layout of plant and equipment. In many cases no further extensions were needed, ample space being available for post-war projects; in others, however, extension schemes were put into operation, the more important of which are briefly referred to in this article.

THE common heavy metals have been known since antiquity and can look back on a thousand years of development, but aluminium has only been obtained in its metallic state from its compounds during the last century, at first in the laboratory and later on a commercial scale. In this country the commercial production of aluminium may be said to date from 1858, when the first aluminium works was established at Battersea by F. W. Gerhard. Upon this, and also upon the fact that the British Aluminium Company was among the very first producers of aluminium by the Hall-Héroult electrolytic process, this country may claim to have one of the longest established aluminium industries in the world.

From the beginning, expansion of aluminium production in this country has been steady rather than spectacular; a slow and deliberate expansion policy was followed, which, by 1938, had brought us to ninth place in the list of the world's aluminium producers while ranking third among the consumers. Production in other countries increased more rapidly than in Britain, mainly because of easy access to high-quality bauxite and to water power resources, which are severely limited in this country.

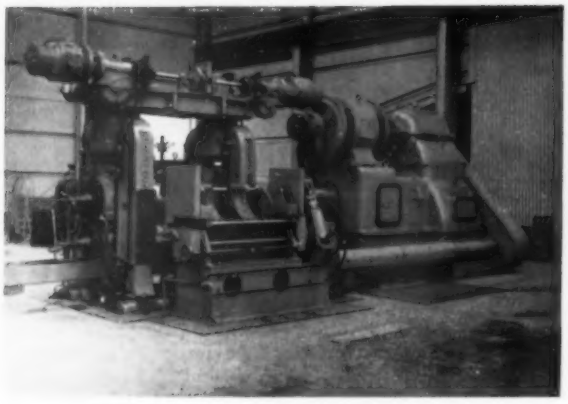
By comparison there was rapid and vigorous expansion of the fabrication side of the industry. Based from the beginning on scientific research, from which was discovered the effects of alloying the aluminium with other metals to produce strong light alloys, which gave new life to engineering development, the material became closely allied to the aircraft and automobile industries, for which scientific control became an essential structural factor. From the research laboratories, growing rapidly with the industry, came a steady stream of improved alloys and techniques to fulfil the ever increasing demands of engineers for stronger and still stronger alloys to enable them to build more powerful engines and lighter and stronger airframes.

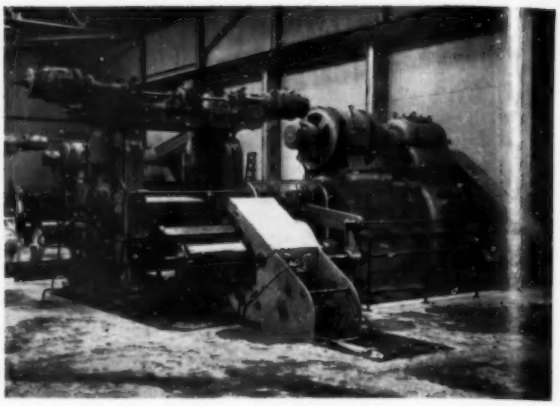
Up to the years immediately before the war the aluminium industry had grown rapidly enough to fulfil all the requirements of the aircraft and other industries for its products. When war broke out this country had a fairly large fabricating industry, adequate in many branches to cope with the aircraft production programme, but with a domestic production of primary aluminium far below requirements. Some developments were made to increase production, but they were on a relatively small scale, and the country became dependent on imports for the bulk of its requirements. Even this source was limited and the Government was forced to advance capital to Canadian and United States producers to enable them to increase their outputs in order to supply



Courtesy of Northern Aluminium Co., Ltd.

A general view of the new semi-continuous mill buildings at Rogerstone which house plant for hot-rolling and cold-finishing aluminium and alloy sheet.





Courtesy of Aluminium Corporation, Ltd.

Part of the rolling-mill plant scheme at Dolgarrog.

our growing demands. For a time we experienced a gap in our resources and frantic efforts were made to ensure supplies; readers will recall the spectacular appeals for pots and pans, but one of the main factors which enabled the gap to be filled was due to the foresight of the secondary metal refiners who had built up considerable stocks of material which they patriotically threw into the pool. Although supplies of aluminium were, at times, very low, and it had been necessary to eliminate practically all non-aircraft uses of the metal, they never completely dried up. In due course the huge new plants in North America came into production and, despite the submarine menace and loss of much aluminium from this cause, the supply difficulties were overcome.

While primary aluminium was thus ensured by imports, fabricated components could not be imported, fortunately, however, the fabricating capacity and technical ability at the outbreak of the war was of a high order and although apparently insurmountable difficulties were encountered by the greatly increased demand, due to the foresight of a number of enthusiasts in the industry in increasing capacity and in the detailed planning of further expansion, the fabricating industry managed to keep abreast of aircraft production programmes, despite the high cost in man-hours of equipment it was necessary to instal.

To increase the output of fabricated components and to continue to increase was of primary importance and, in spite of many difficulties, existing plants were extended, new plants were built, in widely separated parts of the country, equipped and brought into operation in very short times, and records established. Parallel with the expansion of plant were intensive training schemes in moulding and coremaking, press and drop-stamp operations, inspection, and even die-sinking, to fit personnel for the new works. Great changes had to be made in manufacturing methods, procedures based on years of cautiously acquired experiences were altered overnight in the race for production. That quality did not suffer, that few projects met with failure is proof of the great fund of metallurgical knowledge and experience built up in the industry by the long adherence to its policy of scientific research and control.

As production got into its stride demands for fighter aircraft of higher performance became incessant, this imposed demands on the industry to supply aluminium alloys of even higher quality and for increased strength at atmospheric and elevated temperatures. Still in the throes of expansion, the industry intensified its research activities and, without interference with production, alloy compositions were modified, new alloys were developed, and improved methods of casting, working, heat treatment and control were evolved and introduced into production smoothly, without any faltering of the expansion programmes.

Among the many achievements during this period was the revolutionary change in the policy of utilisation of secondary aluminium, to which brief reference has already been made. The technique of transforming scrap of all kinds into high quality material of closely controlled composition, made tremendous advances and the fabricating industry found that, for many components, it could employ proportions of secondary to virgin material that were unheard of in high quality material before the war. Collaboration contributed greatly to this achievement. Users of aluminium alloys made a practice of segregating their scrap according to specification, thus removing much of the tedious work of sorting and blending for remelting, the secondary metal manufacturers greatly increased their plants and instituted the closest control and supervision at all stages in their refining processes. Aluminium founders and fabricators studied the effects of increasing proportions of secondary metal on the quality of their products and evolved safe limits not only for their output generally, but for individual processes and components. The intrinsic value of scrap aluminium alloy is now fully appreciated and it bears the same economic relationship to virgin aluminium as any other scrap does to its parent metal. As during the war years, so to-day, it is vitally important to ensure that every pound of aluminium that can be reclaimed and used again should be recovered.

War conditions thus caused development to proceed at such a high rate that production far exceeded pre-war requirements and considerable development in the application of aluminium and its alloys for peaceful purposes was necessary when the war came to an end, to absorb the increased production capacity available and to make use of the trained personnel that had been engaged on war production. Apart from the aeronautical branch of engineering, the industry was called upon to make almost a new start. Fortunately, due to the foresight of some of the industry's leaders, plans had been made while the war was still in progress, which, while retaining flexibility, enabled the industry to make a rapid change to meet peace-time needs. In addition,

however, the use of aluminium alloys for such severe service conditions during the war favourably impressed engineers and designers and was a great help in the transition from war- to peace-time production.

Apart from the domestic field which offered one of the quickest means of marketing aluminium products, the possibilities in the field of transport, on machine tools, packing machinery and textile machinery, and in architecture and building were investigated. Ventilating ducts and plumbing systems in building development, pre-fabricated building units, heating equipment, doors, windows, and refrigerating plant all provided profitable uses for this material. In marine engineering, shipbuilding and railway engineering, only the fringe of potential applications had been touched in pre-war days. Increased applications in the electrical and chemical industries were investigated and gradually the wider use of the material for packaging—from foil to larger containers—began to be realised.

The aluminium industry has always adopted a far-sighted policy of research and development and, while many problems still confront it, the widespread appreciation of the varied and special utility of aluminium, which has been one of the most compelling forces in its general market expansion since the war, is, in no small measure, due to the continuance of this policy of research and development.

So much new plant was put into operation during the war under the stress of the abnormal conditions then prevailing, that further developments in this direction might reasonably have been expected to have been halted, but this was not the view of the industry. It will be appreciated that at almost every works, at which the war efforts were concerned with aluminium products for aircraft, the changeover to meet the demands of post-war industry necessitated many re-adjustments and alterations in utilisation and sometimes in the layout of plant and equipment. However, in a number of instances, reconstruction and extension schemes have been in operation for some time and new plant installed or in course of erection, not only to increase fabricating capacity, but also the production of the metal.

Of these more recent developments which have taken place, or are in progress, of great importance are the programmes of extension and modernisation at the Newport and Burntisland alumina works of the British Aluminium Company. The main objects of this work are to ensure adequate supplies of alumina for the reduction works in Scotland and, by increasing efficiency, to bring about a reduction in production costs. This work should be completed in 1951. In addition, extensive re-equipment is in progress at the reduction works. At Kinlochleven, for instance, the programme of reconstruction is nearing completion. Considerable progress has also been made at this Company's larger reduction works at Lochaber, Fort William, where reconstruction will be well advanced by the end of this year, completion being expected in 1951. This reconstruction work will not only bring about more economical operation but will increase the production of virgin metal.

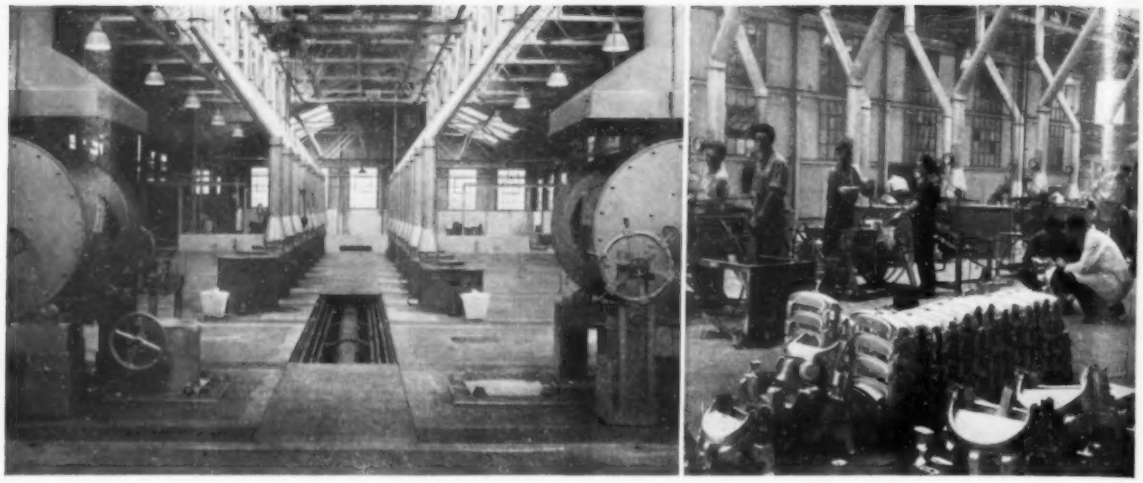
Of considerable importance also are developments in progress on the refining of scrap aluminium. The recovery of this scrap for use with or instead of virgin metal is sound economics and in accord with the general principle of conserving natural resources. It is noteworthy, therefore, that new processes for refining aluminium scrap have engaged the attention of International Alloys, Ltd. for some considerable time. The work has led to the development of a process, which, if successful, will not only allow the production of very pure aluminium from alloys of any description, but might also be developed into a new method for the production of aluminium from ore. Carried out since 1946 at the laboratories of Fulmer Research Institute, Ltd., the work on the project is sufficiently advanced to proceed from the pure laboratory stage to a larger scale laboratory plant, which has aroused considerable interest.

While improved facilities for the production of aluminium, whether primary or secondary, are of vital importance to the aluminium industry, the fabrication section of the industry has not been overlooked. Considerable progress, for instance, has been made in providing increased facilities for the production of tube and sections, but due to the heavy demand for strip and sheet, it is not surprising that new equipment to meet this has received special attention. Of particular interest is the installation of new strip-rolling plant at the Dalgarrog works of Aluminium Corporation, Ltd., which is making good progress. Two "Robertson" non-reversing mills are already erected together with the necessary ancillary plant, including coil annealing furnaces, slitting machine and Halden shears. A third mill is due towards the end of the present year and these three mills will result in an appreciable increase of productive capacity. The mills are being housed in a new steel frame building covered with "Cellastite Sheeting."

The British Aluminium Company has a large extension in hand at its Falkirk works, the greater part of which will be completed about June, this year, full production being expected towards the end of the year. This extension, which should provide employment for a further 200–300 workers, involves new factory buildings covering several acres and comprising two main 100-ft. span bays, and other bays of smaller span. The most

Courtesy of The British Aluminium Co., Ltd.

A general view of the strip mills in production, at Falkirk, with the Davy-United breaking down mill in the background.



Courtesy of High Duty Alloys, Ltd.

Sections of the new gravity die-cast foundry at Slough, showing two Ashton semi-rotary furnaces serving a battery of 18 Morgan bale-out furnaces.

noticeable feature of these buildings is the use of corrugated aluminium for the roof and side sheeting. Here, again the principal items of plant to be installed in these extensions consist of rolling mills, the purpose of which is to increase the Company's capacity for the production of thin gauge sheet and strip for box making, canning and packing, foil stock, etc.

Since the cost of materials is so important a factor in their choice, in order to assist in developing the uses of aluminium and to stimulate demand The British Aluminium Company has decided to pass on to consumers the reduction in rolling costs that are expected to be achieved with the new equipment and consequently the prices are reduced for the thinner gauges of pure aluminium and B.A. 60 ($1\frac{1}{4}$% Mn) alloy sheet, coiled strip and circles. Prices of Rigidal corrugated sheet have also been reduced.

An ambitious scheme is nearing completion at the Rogerstone works of Northern Aluminium Co., Ltd., a semi-continuous rolling mill. This plant's official output is 50,000 tons a year, though the scheme is capable of extension to an annual output of 150,000 tons, if required. Basically the plant comprises hot-rolling and cold-finishing lines, with flattening, shearing, corrugating, temper-rolling lines, etc. The output from this plant is likely to be container sheet for the metal box trade, and heavier gauge material for motor body panels and general applications. The completion of this scheme has been delayed, mainly as a result of the low priority which had been given to plant ordered, however, it is expected to be in operation about the middle of this year.

Towards the end of 1948 a new gravity die-casting foundry was put into operation at the Slough works of High Duty Alloys, Ltd., which is of more than ordinary interest. The object was to meet the demand for high strength aluminium alloy die-castings and the scheme necessitated re-equipping the die-casting foundries for the production of Hiduminium die castings. The opportunity was taken to design this foundry on advanced lines, as will be seen in the accompanying illustration. In the layout, arrangements were made for the adjacent fettling shops and to facilitate the easy flow of castings from the die to final inspection. For this purpose the design included a system of underground trenches along which castings are transported out of the foundry proper. These trenches are covered flush with the foundry floor by cast Hiduminium-chequered plating, ensuring a clean open floor in the foundry. This innovation, together with the exhaust system for the removal of gases from the bale-out furnaces, assist in combining speed of production with cleanliness and comfort for the operators. The aluminium alloys are melted in bulk in two semi-rotary furnaces made by Ashton, Ltd. which service the batteries of Morgan bale-out furnaces, in which the metal is cleansed preparatory to casting.

A further indication of progress in the die-casting field is the scheme of reorganisation nearing completion at the Handsworth, Birmingham works of Northern Aluminium Co., Ltd. This scheme, which will, in effect, double the output capacity for aluminium alloy die castings, expected to be the largest of its kind in the country, is likely to be in operation very shortly.

Aluminium Sheathed Power Cable

It is not intended to refer here to the many fields in which aluminium and its alloys have been accepted in competition with other materials, but an exception can be made for recent applications in the electrical industry which are not only of interest but show the trend of progress. It will be remembered that aluminium conductors were used to a considerable extent before the war. These were the solid, stranded, and steel-cored stranded types, of which the latter is more generally employed to-day; more than 24,000 miles of which have been erected as overhead lines. A more recent application, however, is the use of aluminium as a sheath for the power cable. Intensive research has been carried out by such firms as British Insulated Callender's Cables, Ltd. and Pirelli-General Cable Works, Ltd. with this in view, as a result of which many of the application problems have been solved, and aluminium-sheathed cables are now available in a range of sizes. The design in fittings and accessories has been thoroughly explored, and continued research is making it increasingly evident that this new departure in cable practice will prove of immense value, both technically and economically.

Another method of aluminium sheathing of electric cables has been patented by Standard Telephones and Cables, Ltd. and S. E. Mayer.* The aluminium sheath is first produced by extrusion as an over-size tube. The insulated core is fed into the extruded tube through the hollow mandrel passing through the billet container of the extrusion press. The hollow mandrel is provided with a water jacket to prevent damage to the insulation; thus the core does not come into contact with the inner wall of the aluminium tube until the temperature is such that no damage to the core can occur. When the tube has travelled sufficient from the extrusion die and cooled to a safe temperature, it enters and is drawn through a die smaller than the outside of the extruded tube to form a closely fitting sheath on the cable core.

In connection with this same application the General Electric Company have introduced a machine for the production of aluminium-sheathed cable on a commercial scale, incorporating their recently developed cold-pressure welding process. With this machine cable sheathing can be divided into four stages: flange forming; preparation of flange surfaces; introduction of cable and formation of tube; pressure welding and removal of flanges.

Aluminium strip of the required width is drawn into a pair of rolls from a feed spool at the rear of the machine. The strip emerges as a trough with two horizontal flanges and passes to two rapidly rotating steel scratch brushes, which give the machine surfaces the necessary preparation to enable the cold weld to be made. There is provision at this stage for exhausting the metal dust. The cable and the incomplete sheath are thus brought together and passed through two horizontal tube-forming rollers to the welding rollers, where the projecting flanges are welded together and the excess metal trimmed off. In this machine only the two welding rollers are driven, all the forming rollers running idle.

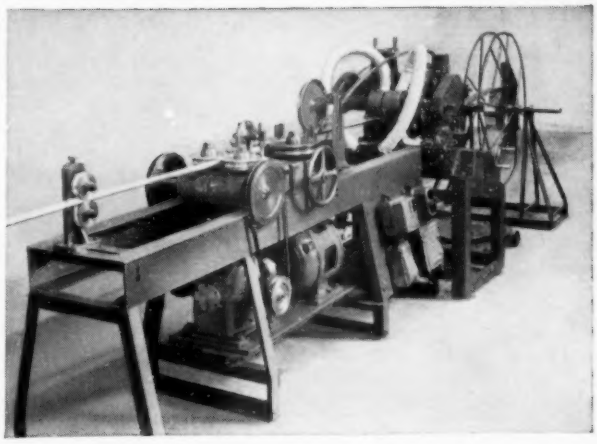
Courtesy of British Insulated Callender's Cables, Ltd.

A stepped sample of a 4-core 660 v., 0.20 sq. in. aluminium sheathed power cable.

The fact that each incursion into new fields of industrial application is, almost invariably, preceded by much circumspection and careful analysis of the problems to be encountered, and many exhaustive tests to determine the best alloy for the work, is all to the good and one of the main reasons for believing that the future holds promise for such a live and progressive industry, although the need for a greatly increased virgin metal production within the confines of the United Kingdom is an ever-pressing one if costs are to be substantially reduced.

Of growing interest in the electrical industry is the use of aluminium conduit. The smooth interior surface of the conduit greatly facilitates cable drawing and its use is claimed to have advantages from a labour-saving point of view. Probably the largest installation of aluminium conduit is that at the new Rogerstone factory of Northern Aluminium Company, where over a hundred miles of aluminium conduit, varying from $\frac{1}{2}$–$4\frac{1}{2}$ in. in diameter and weighing over 80 tons, have been used. The conduits are produced on extrusion presses in an adjacent factory on the Company's 100-acre site, and are supplied in long lengths, sometimes 30 ft.

* British Patent 631, 127.



Courtesy of The General Electric Co., Ltd.

The Pirelli-General cold-pressure welding machine for aluminium sheathing of power cables.

As a rule, cutting and bending aluminium conduit is easier than with steel. Sizes up to $1\frac{1}{4}$ in. diameter are readily sawn without the aid of a pipe vice and can be set over the knee, a great advantage when it is being used in positions almost inaccessible. There is a risk that excessive local movement of the conduit during setting may cause kinking or rippling; to reduce this possibility a somewhat different technique is adopted than is customary for steel. For setting small tubes by hand force should be applied carefully, gradually "inching" along the tube so that the bend is formed progressively. Various types of bending equipment are being successfully used to produce curves quickly; one machine enables a right-angle bend in a $2\frac{1}{2}$-in. diameter conduit in 3 minutes, and for sizes below this handling times are less. For tubes of $\frac{1}{2}$ and $\frac{5}{8}$ in. and those over $1\frac{3}{4}$ in. diameter aluminium is cheaper than steel, while intermediate sizes cost about the same. Aluminium can be set to smaller radii than steel, there is, therefore, less need to use special bend fittings.

Tests on installations have indicated that, in spite of the oxide film, the resistance of screwed joints in aluminium conduit does not tend to increase to any greater extent than in steel systems, and the superior conductivity of aluminium ensures effective earth conductivity. No special precautions have been necessary at Rogerstone in making the screwed joints; no protective coating has been used, except when the conduit is laid in wet concrete when a coating of bituminous paint is applied.

Aluminium Schools for Australia

Earlier this month, 25 of the 100 "Bristol" aluminium schools, recently ordered by the Government of Victoria, Australia, were despatched by rail from the Weston-super-Mare factory of the Bristol Aeroplane Co., Ltd. to Avonmouth Docks, for shipment to Australia. The 44-wagon train-load was most impressive and indicated the importance of this field of application for aluminium.

The Place of Aluminium in Packaging

By Dr. E. G. West, F.I.M., F.R.S.A.
The Aluminium Development Association

The decision to hold a National Packaging Exhibition in London next year is an indication of the increasing interest being taken in this subject as its importance becomes more widely appreciated. Although aluminium has been used in packaging for many years there has been a considerable expansion in this field since the war. The purpose of this article is to summarise present applications and future prospects.

ALUMINIUM has been used for more than 20 years in the packaging industries which have themselves been subject to considerable change and expansion within that period. Tastes and habits have altered, methods of selling have undergone fundamental changes, the demands of large war-time forces in all parts of the world and the re-arrangement of food supply channels due to rationing have all contributed to—and continue to affect—the packaging of goods of all types and sizes. There is strictly no "Packaging Industry" in the sense that there is an "Aluminium Industry," but the necessity of packaging makes itself felt in almost every manufacturing trade and field of commercial activity.

Before the recent war aluminium was gradually becoming known in several forms of "packet," but developments in this field had to be suspended, owing to the demands of the aircraft industry, in almost every country of the world. Aluminium was used to a limited extent for the packaging of certain war stores and, in such countries as Switzerland, for new domestic packaging purposes. Shortages of conventional materials, such as tinplate or timber, gave aluminium the chance of many a trial which might otherwise have been postponed for decades, and it is significant that, to-day, aluminium is holding its own against the former materials in many instances where the shortage factor is no longer important.

The present purpose is to summarise the principal outlets for aluminium in packaging both at present and in the future.

Types of Package

Packaging has been taken to include foil for direct wrapping and for lining of tea chests, etc.; composite foil-organic material laminates; bottle closures; cans, boxes and crates of all shapes and sizes; collapsible tubes; kegs, barrels and drums; and a multitude of those decorative features applied to "packets" of every kind. Among the newest types of package suitable for aluminium are the "envelopes" used in the conveyance of aluminium sheets, and the wrapping of frozen foods.

Requirements of a Package

Packages must be strong enough to carry the load, including impact stress, without the risk of damage to the contents. The material contained in the package must not attack the packet which, in turn, must not contaminate the contents. A package must be readily made, filled and closed, and should be of low weight in proportion to the material packed. The packet must be sufficiently permanent for the period of storage envisaged, plus a margin for abnormal conditions, and, in many cases, it must possess initially, and retain, its appropriate "shelf appeal." Some packets require to be decorative or capable of decoration which will not deteriorate until,

Courtesy British Hygienic Containers, Ltd.

"Alplex" food containers are a well-established application of aluminium in packaging.

Courtesy Gee Manufacturing Co., Ltd.

Motor oil containers made of cardboard covered by aluminium foil.

Courtesy Aluminium Laboratories, Ltd.

A selection of shallow drawn aluminium containers, pressed from half-hard commercial purity sheet 0.014 in.thick.

Courtesy Aluminium Laboratories, Ltd.

This drawn aluminium can is used unprotected for sweetened milk and anodised for milk without sugar.

at least, the goods are sold. Finally the cost of the package must bear the correct relationship to the price of the contents, taking into account such factors as publicity value, re-use possibilities, return and recovery or scrap value, and availability in the required quantities.

Characteristics of Packaging Material

It is now possible to consider briefly the desired properties of a packaging material. First, the strength must be sufficient; secondly, the contents of the pack must not attack the material with which it is in contact; then, it must be amenable to the manufacturing methods available. The cost must be low and the quantity adequate for all foreseeable demands within the requisite time period. Methods of finishing for protection or decoration should be readily applicable and should be inexpensive.

Aluminium and certain aluminium alloys fulfil many of these requirements, and possess the advantage of low weight by comparison with most competitive materials. There are, of course, problems still to be solved in this field of development—as in many others—but the researches in progress in both the aluminium-producing and aluminium-using industries are progressing towards complete answers to the questions which demand investigation. Results to date in this country and abroad, indicate clearly that aluminium has a sufficiently useful combination of properties to allow it to expand its present usage many times in packaging.

Aluminium itself is largely used for many packaging applications owing to the ease with which it may be worked, the variety of finishes which can be applied and its high resistance to corrosion. Some of the alloys are increasing in their application to packaging and, in particular, the low alloy content materials such as the $1\frac{1}{4}\%$ manganese alloy or aluminium-magnesium alloys containing up to 2% magnesium are particularly valuable when rather higher strength than that of the pure metal is required. For the larger sizes of container, of the box type, and vessels to hold liquid, such as milk churns, the heat-treated aluminium-magnesium-silicide alloys are used and there has also been some usage of the high strength duralumin-type alloys for large road-rail containers and the like.

Hygienic Requirements.—A question frequently asked when discussing aluminium as a packaging material for food products is, of course, the possible risk of contamination of the food by the metal. A very large number of tests and a great deal of experience have all shown that the metal is not only inert to the majority of foods with which it is in contact but, also, that any aluminium entering the food is harmless and does not have any toxic effects on the human being. Doubts are sometimes expressed on this point by the uninformed and it is of considerable value to the aluminium and food industries that a thorough investigation of the evidence was carried out by one of the D.S.I.R. organisations and the results published in Special Report No. 50, "Aluminium and Aluminium Alloys in the Food Industry" (H.M. Stationery Office), 1948. Reference in this connection should also be made to the recently published book—"Canning Technology," by Howard (J. & G. Churchill, Ltd., 1949), wherein the toxicology of aluminium in canning is convincingly discussed.

Foil

Aluminium foil is commercially pure aluminium of 99% purity or higher, rolled to thicknesses from 0·00025–0·005 in. For certain purposes foil of high purity is necessary in order to allow easy wrapping without any spring-back. Freedom from impurities is also of great importance as the thickness is reduced, otherwise pin holes may occur to the detriment of the contents of the pack. Moisture is transmitted through such pores or pin holes, and in this connection it may be noted that a recent development is to use two foils together, each being much thinner than a single foil used for the same purposes. The resulting foil laminate is much more ductile than a similar thickness in one piece, allowing close folding to be carried out. Resistance to perforation on folding is also greatly increased and the rate of moisture transmission virtually zero.

Foil production is carried out by specialist concerns employing precision equipment to give foil of uniform thickness, free from pin holes, buckles or waviness, and

possessing a bright surface finish. Final reduction is therefore carried out on highly-polished rolls from which all dust must be completely excluded. Continuous lubrication of the rolls is necessary by means of suitable rolling oils usually consisting of mixtures of light mineral oils with stearine. Thickness is controlled not only by rolling pressure but also by the tension on the strip fed into the mill. The final pass is given by rolling two thicknesses together to give a bright surface on the sides in contact with the rolls and a somewhat duller surface on the inner faces of the foil. The coils are rewound to separate them and annealed for comparatively long periods, such as 8 hours at temperatures around 400° C., in order to soften them fully. Degreasing may be carried out before annealing but the thin film of rolling oil is readily removed in the annealing process.

Usually plain foil for packaging is less popular than foil decorated either by embossing or coloured printing or, more often, a combination of printing and embossing or other surface treatment. Problems involved in securing adequate adhesion with rapid printing and quick-drying have been largely overcome and the range of decorative effects which can be achieved, by suitable combination of inks and surface finishes ranging from high reflectivity to opaque matt, is extraordinarily wide.

In addition to printing or lacquering for decorative purposes, it is often necessary to apply a protective lacquer to foil in order to prevent direct contact between the metal and the contents of the package for such packs as portions of processed cheese. Wrapping in plain or lacquered foil is but one means of using this material in packaging. It may be readily pressed, either alone or in combination with paper, to form containers for ice cream, fruit jellies, trifles and the like, as illustrated. Another well known use of foil is for milk-bottle tops and some 2,000 tons is used each year for this purpose. The advantages of aluminium for this application include not only its impermeability to moisture or substances which might contaminate the milk, its cleanliness, smooth and hygienic surface, but also its ease of fitting and removal combined with the more readily cleaned bottle neck as compared with the type requiring an inner ledge to take a cardboard closure. Pleated or crimped foil, usually highly decorated, is also familiar as a cap on bottles containing wine and similar liquids. A recent development is the wadded bottle closure of appreciable thickness used for capping milk that is sterilised in the bottle, and as an alternative to tinplate for beer-bottle tops which do not require a special instrument for opening.

Many other applications of aluminium foil have been opened up by combining it with other materials such as paper, cardboard, plywood or plastics to give adequate strength for heavier duty than foil alone could carry. Laminates of this kind often involve the use of adhesives which must be harmless to the metal, quick drying and easily applied. Both water soluble and plastic adhesives have proved suitable, but the chloride content of the paper or board must be kept below 0·07%, otherwise corrosion of the foil may occur, as paper normally absorbs moisture from the atmosphere to the extent of 5–8%. One of the latest developments involves the composite plastic-backed foil which can be heat-sealed to give a completely moisture-proof bag. Foil-backed cardboard has been used in the form of bottles to contain many liquids such as oil. One of the oldest uses of aluminium foil is, of course, for lining tea chests and this has been followed by containers in which the foil is firmly stuck to plywood which may be moulded to special shapes after cutting. A particularly interesting application of aluminium foil depends on the cathodic protection of bright steel articles wrapped in foil. Surgical instruments, precision machine parts, bearings and the like are coated with an inert oil and closely wrapped in foil. Any moisture retained in, or entering, a package does not immediately cause rusting of the ferrous material but preferentially attacks the aluminium, so protecting the articles concerned.

The use of aluminium foil for wrapping cigarettes and tobacco, for a wide range of chocolates and other confectionery, and for various toilet preparations, has gradually returned since the end of the war, and it is likely that a considerable expansion in the output of foil may become necessary as the general supply of goods improves and as the public become more conscious of the advantages of this method of ensuring hygienic handling.

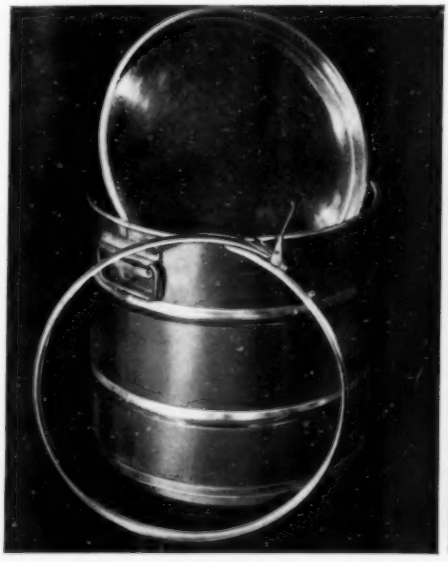
Collapsible Tubes

Although collapsible tubes have been used for over 100 years, for example, for artists' colours, aluminium is a comparative newcomer to this field. They are made by impact extrusion and until recently the materials were confined to tin or lead or tin-lead alloys, but the use of pure aluminium, introduced in part as a temporary measure, has expanded and is likely to continue on an increasing scale. The metal cost of aluminium is lower than that of tin or lead to-day, but the cost of manufacturing aluminium tubes is somewhat higher as it is necessary to anneal them to obtain the desired degree of pliability. With the change-over to aluminium it became necessary in many instances to line the inner surfaces of the "as-extruded" tubes either with lacquer or with wax. The development of successful lining techniques has greatly increased the field of application of aluminium collapsible tubes and is a vital factor in their continued use and expansion. It will be appreciated that the problem is extremely difficult owing to the great care with which the empty tubes must be handled at every stage. Slight damage before filling may be responsible for a costly hold-up in the operation of the high-speed equipment and numerous ingenious devices have been developed for lining the tube. The anti-corrosive coating is applied sometimes before and sometimes after printing or otherwise applying the required decorative finish. Wax appears to be the most suitable lining material as it is not susceptible to any ageing and it adheres better than the usual stoving lacquers. The slugs or blanks must be of dead-soft metal and free from any burrs or hard spots from the blanking operation. Attempts to use metal of comparatively low purity or aluminium alloys have not proved successful. The range of materials packed in aluminium tubes is wide and includes many medical and pharmaceutical preparations, tooth pastes, printing ink, shoe cream and other cleaning materials, foodstuffs—for example condensed milk and mustard, on the Continent—and large tubes containing lubricating oil and greases.

Cans and Pressed Containers

The entry of aluminium into the can- and box-making fields has been by no means simple for, apart from the lack of experience of aluminium in contact with many packs, the methods of manufacture suitable for aluminium cans differed fundamentally from the estab-

lished processes developed for tinplate and lacquered steel containers. The biggest single difficulty has been in making joints for which soldering is so largely used in the case of tin. Various forms of mechanical seam have been developed for lacquered steel containers and some of them are suitable for aluminium cans which are made airtight by some form of plastic sealing strip or ring. Modern can-making machinery, designed in accordance with the requirements of tinplate-can manufacture, cannot be readily adapted to suit aluminium and this has undoubtedly accounted for much of the reluctance on the part of can makers to effect a change.



Courtesy Morrison's Engineering, Ltd.

Five-gallon drums for edible oils, fats and fruit juices. The lids are provided with a gas-tight seal.

Impact extruded aluminium tubes are widely used on the Continent for such food items as condensed milk and mustard.

The output of cans in Great Britain totals some 1,800 million per annum, and aluminium so far has been used for only a small proportion of this total. Cans and boxes can be broadly divided into four groups—namely, those made as (1) shallow pressings; (2) deep-drawn articles; (3) impact extruded containers; and (4) folded-type or built-up boxes or cans.

The shallow type of pressed container, as used for certain fish packs, dentifrice powder, meat and fish pastes, are comparatively simple pressings made from 99·5% pure aluminium or occasionally the aluminium-1¼% manganese alloy. The metal thickness ranges from approximately 0·01–0·02 in., the amount of metal being kept to a minimum in order to maintain the lowest price consistent with the required quality and robustness. The methods of protection are fairly well established and, in general, a sheet of special drawing quality is required. Deep containers necessitate inter-stage annealing with the usual precautions to avoid excessive grain growth leading to " orange-peel " effects.

The shallow drawn or pressed container is used for fish packs and in at least one instance protection against the contents is given by anodising the sheet before drawing. Continuous anodising plants for coiled strip are a practicable proposition and allow low overall costs to be attained. For many packs, particularly dry goods, tobacco, etc., no protection is required, but for packs containing acids or salt, lacquering is sometimes preceded by anodic oxidation or a surface treatment of the " M.B.V. "-type. Drawn aluminium cans require the lacquered coating to be applied after drawing, although shallow cans may be manufactured from lacquered sheet or strip. It has been found that the lacquers developed for tinplate are not usually suitable, without modification, for aluminium, but a certain amount of work has been published recording the results of investigations into the suitability of various artificial resin lacquers, both from the point of view of ease of application and stoving, and taking into account their suitability for contact with foods for appreciably long periods. It has been established that there are several successful types of lacquer available for aluminium, and capable of resisting the acids derived from fruit, vegetables, fish, oils, jam, meat and pickles, as well as foodstuffs containing a high percentage of sulphur.

Impact extruded cans have been made up to about 4 in. diameter but the power required is, of course, a limiting factor on increasing the size of containers made by this method. Again, the container must be coated when necessary after manufacture and annealing may be required to allow sealing to be effected satisfactorily. The necessity for such annealing may depend on the type of seam required and a suitable rubber or plastic sealing ring is, of course, incorporated. Local annealing of the rim may be sufficient and practicable in many cases.

Barrels and Drums

The entry of aluminium into this field has been dictated almost solely by shortages of steel, tinplate and Memel oak for barrels. Kegs and drums are made on the same general principle as that employed for smaller metal containers except that the side seam, and occasionally the top or bottom, are seam welded. Generally, however, ordinary double-seam construction is used and, occasionally, arc welding is used for drums both for construction and for attaching strengthening hoops. A recent development is the use of a collapsible drum which is supplied unassembled and erected when required for filling. The range of products carried in drums is extremely wide—dry and liquid chemicals, dyestuffs, oils and grease of all kinds, resin, spirits, paint and distemper, certain acids and alkalies, lime and similar substances, and vegetable oils and extracts. The sizes range from a few pounds or 1-gallon to 120-gallon drums, and the type of end fitting, lid, filling arrangements, reinforcing ribs or corrugations and other design points

are determined by the service required and whether the containers are returnable or not. The suitability of aluminium to resist attack by many of the products carried is shown by the popularity of aluminium-sprayed coatings on steel as a means of protecting the latter. The higher cost of aluminium, however, has meant that hitherto aluminium drums have been almost always of the returnable variety or used, in general, for the more expensive products packed in this manner. Some of the stronger alloys have been successfully used for drum manufacture and seam welding is possible for heat-treated materials. The argon arc-welding process should prove a satisfactory means of construction for the non-heat-treatable alloys and the method has, in fact, been extensively adopted for the joints in aluminium alloy beer barrels.

Several designs of beer barrel or drum have been mass-produced both in this country and the U.S.A., and according to the experts the metal has no effect on the quality or keeping properties of the beer. Recently aluminium cans have been made for the paint trade and are already proving their worth. Care has to be exercised by the users when aluminium has replaced steel and when both steel and aluminium are returned at the same time for cleaning and refilling. The standard method of cleaning steel drums is usually the use of a caustic soda solution and it is, of course, not suitable for aluminium drums which necessitate an inhibited alkaline detergent or a solvent degreaser. Changes of this kind in handling methods, though they may be small, militate against the adoption of aluminium, particularly when the changeover from steel takes place in a piecemeal manner.

Providing that the proper means of pre-treatment are adopted there is no difficulty in applying any required protection or decorative paint system to the surface of aluminium drums and kegs.

Crates

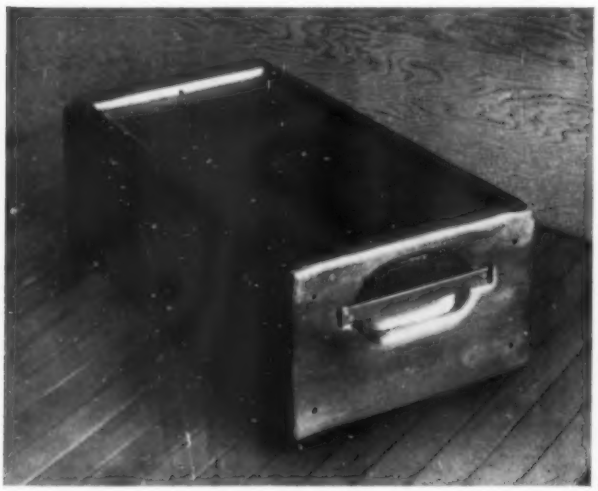
Aluminium alloy crates are gradually taking their place alongside galvanised steel and timber, and the savings due to the lower weight of the light metal article are often able to offset the higher initial cost. Many designs of bottle crate for milk, alcoholic and soft drinks, lubricating oil and similar commodities have been marketed, some of them combining high-strength aluminium alloys and magnesium-base alloy.

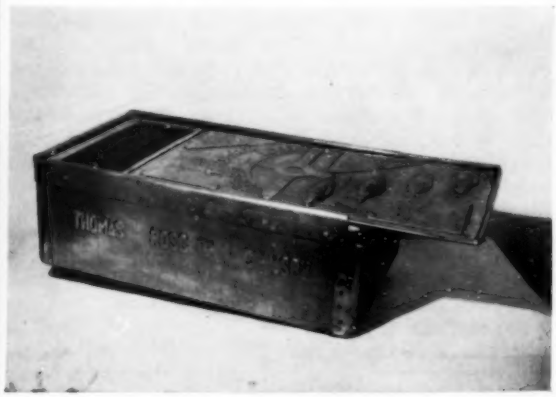
Other types of crate have been developed for the carriage of soft fruits and other agricultural products where, again, the higher initial cost is offset by the longer life and ease of handling. The untreated metal withstands the action of fruit juices, etc., satisfactorily although in some cases a heavy sealed anodic film has proved necessary to afford adequate protection.

A rather similar application is that of fish boxes, which have now proved their worth and have been widely adopted at many of the fish-handling ports. The fact that they can be thoroughly washed to give an almost sterile surface has greatly lengthened the time for which fish can remain packed without deterioration. The absence of smell arising from fish oils, etc., absorbed by wooden boxes makes the new containers popular with those responsible for transporting, storing and distributing fish. A recent development involved the use of aluminium foil-plastic laminates in which fish may be wrapped and packed in aluminium alloy crates.

Finally in this section mention is made of the many road and rail containers capable of carrying loads up to 10 tons which have been used for a sufficient number of years to prove that aluminium is suitable for this arduous service. For example, a road/rail container for fish is able to carry 6 tons, the empty tare being only 15 cwt.

In conclusion it is evident that aluminium on its merits has already been able to compete in many parts of the packaging field with the old-established materials and this trend will continue. Furthermore, as the methods employed by the packaging industries improve, as the necessity or desirability for better packaging becomes more widely appreciated, and as new types of package are developed, the use of aluminium in the form of foil, impact extrusions, drawn or pressed cans and fabricated containers of all kinds should greatly expand. Although during the past year the supply of thin gauge aluminium suitable for metal containers has not quite equalled the demand, new rolling mills and additional equipment are making the supply position easier. When the present programme of building and installation of new plant has been completed it is confidently expected that there will not only be an improvement in deliveries but also a reduction in the price of standard aluminium sheet and strip products for use by the packaging industry.





Two examples of the many types of fish box now in use. They are drawn or pressed and fabricated by welding or riveting, usually in one of the aluminium-magnesium alloys.

Macro- and Micro-segregation in Ingots of Two Aluminium Alloys

By P. Brenner, Dr. Ing. and H. Kostron, Dr. Ing.

An investigation is described which was carried out to determine the micro-segregation of two aluminium alloys and for which a Hanemann micro-hardness tester was used. The main purpose of the investigation was to obtain a general idea of the micro-segregation in the grains at different places in the ingot which show the phenomenon of inverse segregation.

As a result of the changes which take place during the solidification of binary or complex alloys, the local concentration of alloying elements in the solidified ingot may vary considerably from the average concentration.

These changes have already been calculated in detail for a binary system by E. Scheuer.[1] The variations in composition take the form of micro-segregation within the grains and general segregation throughout the ingot. The interior of the grain, which is the first part to solidify, has a different composition from the exterior, which solidifies last. In a similar way there is a macro- or general segregation, throughout the ingot, which is superimposed on the micro-segregation. As a result, the average concentration, calculated for a number of adjacent grains, varies from place to place in the ingot.

In some cases this segregation is simply due to the fact that the first solidified zones of the ingot correspond to the lowest, and the last solidified zones to the highest, concentration: in other cases an inverse segregation occurs in which the areas of concentration are reversed.[2]

To date, with a few exceptions, only macro-segregation has been investigated quantitatively, since the measurement of differences in concentration within the grains is very difficult. On the other hand, macro-segregation in ingots can be determined by chemical analysis—so-called "zone - analysis" — since only the determination of an average value of the concentration is required in this case.

The development of micro-hardness testing, however, now enables simple and accurate determinations of hardness variations within the grains to be made, and in this way the distribution of alloying elements in a single grain can be estimated, since hardness and concentration can be correlated. The instrument used in the investigation described in this article was that due to Hanemann.[3]

The main purpose of the investigation was to obtain a general idea of the concentration of alloying elements in the grains at different places in the ingot which show the phenomenon of inverse segregation. To draw conclusions from the results, it is necessary to assume that the concentrations, calculated on the basis of measured hardness values, can differ to some extent from the concentration existing at the moment of solidification.

Generally speaking, it is a question of the rate of cooling after solidification, and, therefore, of the method of casting, as to how much further levelling of the concentration is possible, by processes of diffusion, after solidification. Certain apparent anomalies in the results can only be explained if a relatively high rate of diffusion in slowly cooled ingots is assumed.

Materials

The investigation was made with ingots of a binary alloy of aluminium (99·99% purity) and 4% copper, cast into ingots of 200 mm. (8 in.) dia. One of the ingots was continuously cast, a short water-cooled mould being used, and the surface of the descending ingot sprayed with water.[4] The other ingot was cast in the usual way in an ordinary thick-walled mould of aluminium.

The results were compared with those on a commercial aluminium-copper-magnesium alloy, an ingot of which was continuously cast. This ingot had a diameter of 220 mm. (about 9 in.) and the composition of the alloy was: 3·82% Cu, 1·08% Mg, 0·75% Mn, 0·75% Si, 0·25% Fe, remainder aluminium.

Testing Method

From the middle part of each ingot a disc of 15 mm. thickness was sawn and the disc radiographed to determine the quality of the ingot. The specimens for micro-examination and hardness-testing were then cut, some from the core and some from the outer zone.

The specimens were examined in the "as-cast" condition as well as after a homogenising treatment at 500° C. for 20 minutes, and 1, 3 and 9 hours, followed by water quenching. The soaking at 500° C. was carried out in a salt bath.

Mechanical polishing with alumina was used for the preparation of the specimens. Buckle[5] showed that mechanical polishing results in cold-deformation of the surface layer, so that the micro-hardness numbers are higher than for undeformed material. This was not considered a serious drawback, however, since the problem was not to determine absolute hardness numbers but differences in hardness in different zones. Before hardness testing, all specimens were etched in Keller's reagent[6] to see where solidification had started and where the last zones to solidify, with the highest concentration, were to be found.

The tests were made with the Zeiss Hanemann-type micro-hardness tester used in conjunction with a Zeiss "Neophot" metallograph. The same load was always used to make the impressions for any series of tests where hardness comparisons were to be made (4·5 g. corresponding to 2·0 divisions of the scale).

The micro-hardness number was calculated from the equation:

1 Scheuer, E., *Z. f. Metallkunde*, 1931, p. 241.
2 Brenner, P. and Roth, W., *Z. f. Metallkunde*, 32 (1940), p. 10.
3 Hanemann, H. and Bernhardt, E. O., *Z. f. Metallkunde*, **32** (1940), No. 2, pp. 35–38.
4 Brenner, P. and Roth, W., *Metallwirtschaft*, **21** (1942), p. 695.
5 Bückle, H., *Zeiss Nachrichten*, **5** (1944), 5, 93–100.
6 Keller, *Mining and Metallurgy*, 1935, p. 573. [Composition of reagent:—0·5 g. NaF, 2 ml. HCl (1·19), 1 ml. HNO_3, 97 ml. distilled water. Etching time—3 minutes].

$$H_m = \frac{2\,P\cos\beta}{d^2}$$

where P = test load (4·5 g.).
β = angle of pyramid (22°).
and d = diagonal length of the impression in μ.

Correlation Between Micro-hardness and Chemical Analysis

It was first necessary to determine the relationship between micro-hardness and concentration by calibration tests. In the case of binary alloys of a certain structure (e.g., homogenised) such a calibration curve can be established without difficulty.

Five alloys of high purity aluminium (99·99%) and additions of electrolytic copper ranging from 0–6·64% were prepared and small sheets, 1·0 mm. thick, were made from these alloys in the usual way. The copper contents were determined colorimetrically after the material had been completely homogenised by annealing at 500° C. for 12 hours, followed by water quenching.

In order to obtain reliable average values, 20–30 micro-hardness tests were made on each specimen. The results showed that, as was to be expected, the hardness number was completely independent of the position of the impression within the grain.

The results of these tests are set out in Table I.

TABLE I

Copper content %	Average diagonal of impression – mm.	Micro-hardness kg./mm.²
0·00	0·018	26
1·50	0·013	46
2·44	0·012	58
4·36	0·010	88
6·64	0·008	122

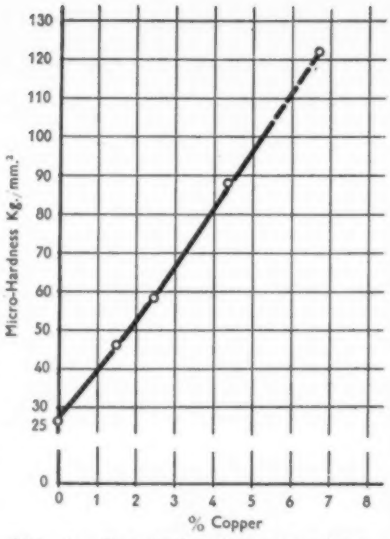


Fig. 1.—Correlation between micro-hardness and copper content (solution heat-treated aluminium—4% copper alloy).

As shown in Fig. 1, there is a fairly linear relationship between the micro-hardness and the concentration of copper. Homogenised aluminium alloys of high purity age-harden considerably, even at room temperature[7]. In the experiments referred to above, this effect was not detected, possibly as a result of iron and silicon being taken up during melting.

For commercial aluminium-copper-magnesium alloys, of course, such a simple relationship cannot be expected, since there are more variables. If the amounts of copper, magnesium and manganese vary at the same time, for instance, it is not possible to draw conclusions concerning a single variable on the basis of changes in hardness. On the other hand, some qualitative evidence on the uniformity or otherwise of the distribution of concentration within the grain may be disclosed. Such data are, for example, sufficient to indicate the levelling of the concentration within a grain during homogenisation. In general, it cannot be assumed that uniform hardness corresponds to a uniform composition in a complex alloy. Such a conclusion may be possible if the hardness values at different points gradually approach a common end-value after homogenisation, since it is known, in this case, that more uniform hardness is brought about by a levelling of the concentration gradients by diffusion.

It must be emphasised that apart from the chemical composition of the material, other factors influence the hardness. One such factor is the type of phases present—e.g., in the binary aluminium-copper system it might be solid solution or solid solution + $CuAl_2$. Other factors such as age-hardening, cold-deformation, etc., also affect the hardness values obtained. The calibration curve shown in Fig. 1 is thus only correct for a solid solution. The alloy containing 6·64% copper cannot correspond to this condition because 5·7% is the limit of solid-solubility of copper in aluminium. In those cases where the limit of solid-solubility is exceeded a pronounced deviation from a straight line must be expected.

Contrary to the aluminium-copper alloy investigated, considerable age-hardening occurs at room temperature in the aluminium-copper-magnesium alloys. It is necessary, therefore, to ensure that only comparable stages of age-hardening are tested.

7 Gayler, M. L. V., *J. Inst. Metals*, **63** (1938), No. 807, p. 67.

Fig. 2.—External zone, unetched. × 100

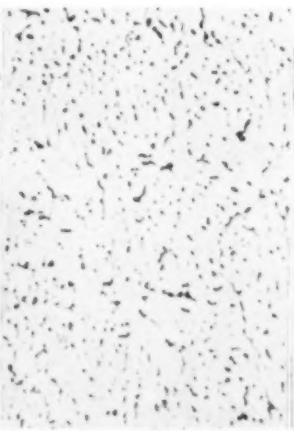


Fig. 3.—Core, unetched. × 100

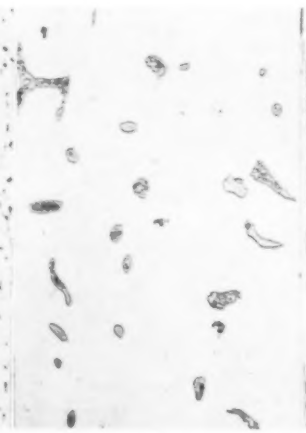


Fig. 4.—$CuAl_2$—inclusions, unetched. × 600.

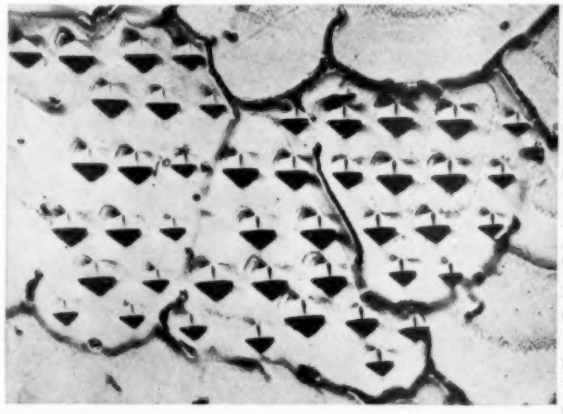
Fig. 5.—External zone, etched for 3 mins. × 100

RESULTS

Aluminium-Copper Alloy—"As-Cast"

Continuous Casting

Figs. 2 and 3 show micrographs of unetched specimens taken from the outer zone and the core of the ingot. The $CuAl_2$ inclusions at the boundaries of the grain-like dendrites appear dark. The grain-like parts, surrounded by these inclusions may be termed "cells." The average distance between these inclusion regions (average diameter of the cells) measures 54 μ in the outer zone and 24 μ in the core.



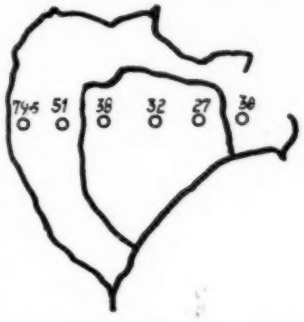


Fig. 6.—External zone. × 500

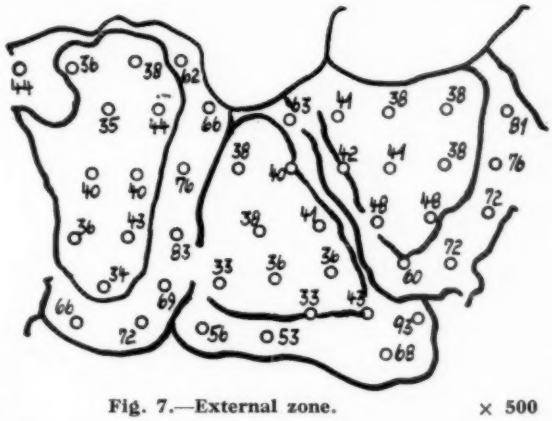


Fig. 7.—External zone. × 500

Figs. 6 and 7.—Micro-hardness tests on an aluminium—4% copper alloy, continuously-cast ingot, "as-cast" condition, etched.

At a higher magnification (Fig. 4) it can be seen that the $CuAl_2$ inclusions form a more or less modified eutectic (*entartetes Eutektikum*) with the solid solution.

After etching, the segregation within the grains is very apparent, since the attack by the etching reagent is largely dependent on the copper concentration (Fig. 5).

Outer Zone.—Fig. 6 shows the variation in hardness along a cell diameter. The region where the growth of the cell started—i.e., at the minimum copper concentration, is clearly shown by the minimum hardness. An intermediate zone, somewhat more heavily etched, separates the soft internal zone, less rich in copper, from the outer zone of higher copper content and greater hardness.

In Figs. 7 and 8 the results of hardness cells containing two dendrites are shown. The average hardness of the inner zone of the cell in Fig. 7 is 37 kg./mm.^{2} and of the outer zone, 70 kg./mm.^{2}, corresponding to copper contents of 0·8% and 3·2%, respectively (see Fig. 1). Fig. 8 shows a primary solidified dendrite, the branches of which are surrounded by areas which solidified last and have a hardness considerably higher than that of the primary dendrite.

Figs. 9 and 10 show details of Fig. 8 at a higher magnification. The primary solidified dendrite has an average hardness of 37 kg./mm.^{2} (corresponding to 0·8% copper) and the areas last to solidify an average hardness of 60 kg./mm.^{2} (2·5% copper).

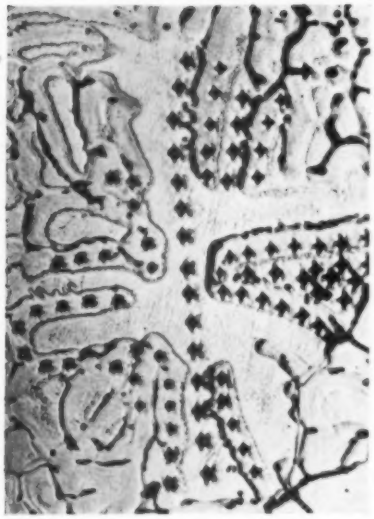
In order to investigate whether the intermediate roughened zone, separating the soft inner- from the hard outer-zone (see Fig. 6), plays a special role in respect of its hardness, tests were made on some of the larger of these zones, Fig. 11. The result was an average hardness of 49 kg./mm.^{2} (1·8% copper) in such a zone, compared with an average hardness of 62 kg./mm.^{2} (2·7% copper) in the outer zones. The roughened area, therefore, corresponds to an intermediate zone, both as regards hardness and copper content, between the primary solidified interior of the cells and the areas which solidified last.

Core.—Since the specimens prepared from the core of the ingot had such fine structures, it was difficult to make a sufficiently clear distinction between the inner and outer zones of the different cells. The results of hardness tests on such a cell are shown in Fig. 12. For the inner zone, a hardness of 70 kg./mm.^{2} (3·2% copper) was found, whilst the outer zone had a hardness of 92 kg./mm.^{2} (4·7% copper). These results indicate that the average copper contents of the cells in the core of the ingot are considerably higher than in the outer zones of the ingot.

Chill Casting

Micrographs of the outer zone and core of the chill-cast ingot are shown in Figs. 13 and 14. The average diameter of the cells was found to be 191 μ for the outer zone and 135 μ for the core of the ingot. These values are much larger than the figures for the continuously cast ingot (54 μ and 24 μ, respectively). Segregation within the cells is revealed by etching in Figs. 15 and 16.

Outer Zone.—A cell with hardness impressions is shown in Fig. 17. As in the case of the continuously cast ingot, the outer zone of the cell has a considerably higher hardness than the inner zone—87 kg./mm.^{2} (4·3% copper) and 61 kg./mm.^{2} (2·6% copper), respectively. It should be mentioned



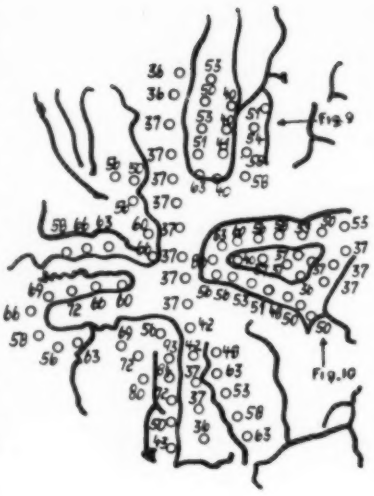
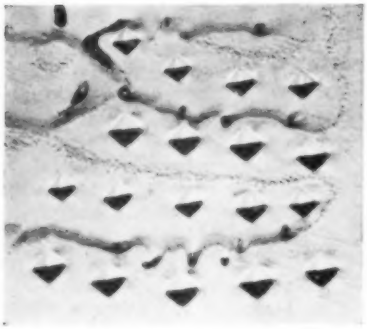


Fig. 8.—External zone. × 200



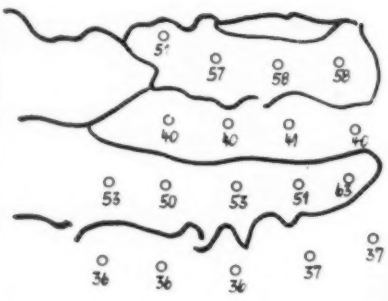
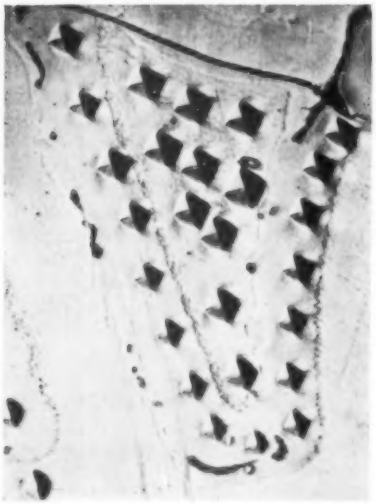


Fig. 9.—Part of Fig. 8. × 500

Figs. 8 and 9.—Micro-hardness tests on an aluminium—4% copper alloy, continuously-cast ingot, "as-cast" condition, etched.



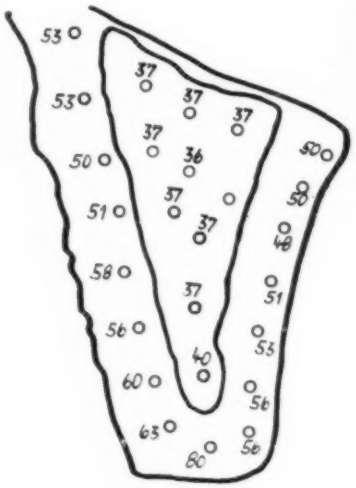


Fig. 10.—Part of Fig. 8. × 500

that only impressions which were not too close to the Cu Al_2 zone were taken into account—otherwise too high a value resulted from the effect of the hard particles of intermetallic compound in preventing flow under the indenter. The hardness of the cell boundaries was between 304 and 414 kg./mm.²

Fig. 18 shows a hardness exploration on another cell. The outer zone has an average hardness of 84 kg./mm.² (4·1% copper) and the inner zone, 52 kg./mm.² (2·0% copper). The substance at the cell boundaries has a hardness of 198–308 kg./mm.² It appeared worthwhile to represent graphically the change in copper content across different sections of these larger cells. This has been done, in Figs. 19 and 20, for two sections through the cell shown in Fig. 17, whilst Fig. 21 shows the graph for one section through the cell shown in Fig. 18. These graphs show the small variation in copper content over a large area in the middle of the cells and the rapid increase in copper content towards the cell boundaries.

Core.—Hardness values for different cells in the core of the ingot are shown in Figs. 22 and 23. The average values are 76 and 75 kg./mm.² (about 3·5% copper) for the outer; and 46 and 42 kg./mm.² (1·6% and 1·3% copper) for the inner zones of the cells.

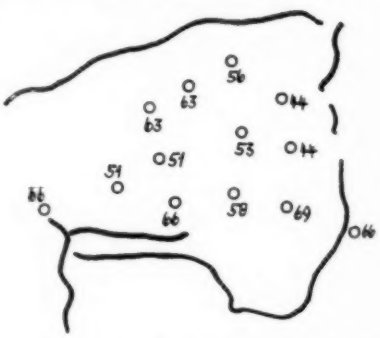


Fig. 11.—External zone. × 500

Figs. 10 and 11.—Micro-hardness tests on an aluminium—4% copper alloy, continuously-cast ingot, "as-cast" condition, etched.

Aluminium-Copper Alloy—Solution Heat-treated

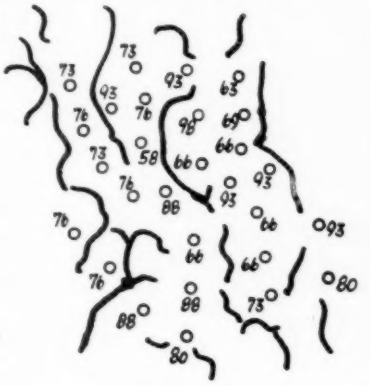
The same specimens of the continuously and chill-cast ingots as were investigated in the "as-cast" condition were solution-treated, as described earlier, in order to see what effect this homogenising treatment had on the concentration gradients in the cells, and, in particular, to determine the time necessary for complete homogenisation.

Continuous Casting

An example of the results obtained is given in Fig. 24. After an annealing time of 20 minutes at 500° C., the average hardness of the outer zone of the cell is 79 kg./mm.² (3·8% copper) and of the inner zone 74 kg./mm.² (3·5% copper). Though a certain amount of copper in the form of $Cu Al_2$ remains undissolved, the results approach the nominal concentration of the 4% alloy.

Chill Casting

Chill castings require longer soaking times at 500° C. than do the continuous castings before homogenisation is achieved, because the larger cells involve greater diffusion distances. This fact is demonstrated in Fig. 25 for a specimen from the core of the chill-cast ingot. After a homogenising treatment of 3 hours at 500° C. the



Core. × 500

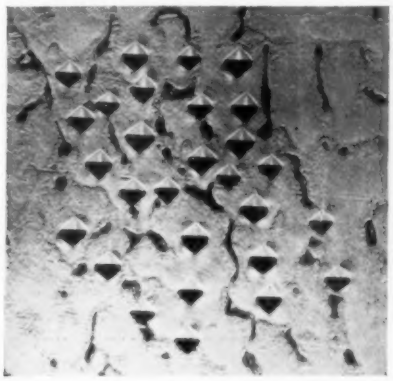


Fig. 12.—Micro-hardness tests on an aluminium—4% copper alloy, continuously-cast ingot, "as-cast" condition, etched.

Fig. 13.—External zone, unetched. × 100.

Fig. 14.—Core, unetched. × 100

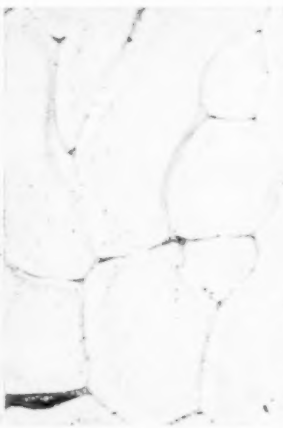
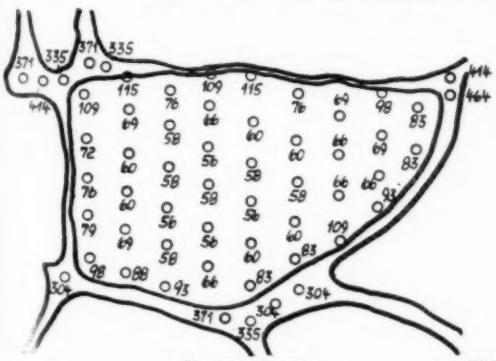


Fig. 15.—External zone, etched. × 100

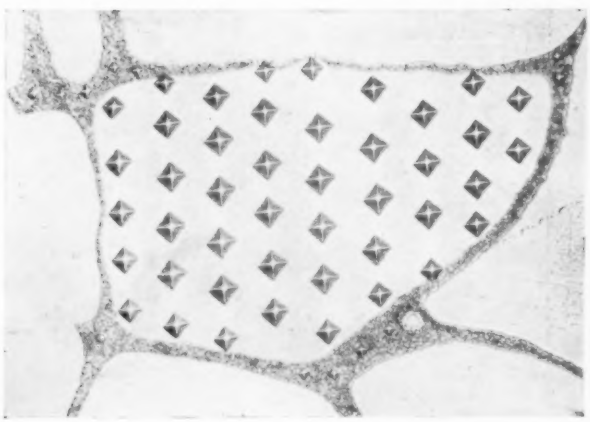
Fig. 16.—Core, etched. × 100

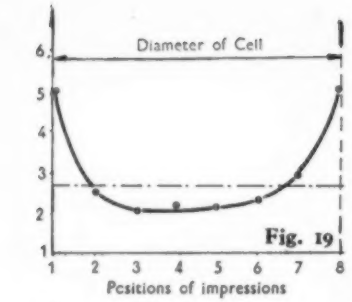
Figs. 13-16.—Photomicrographs of an aluminium—4% copper alloy, chill-cast ingot.

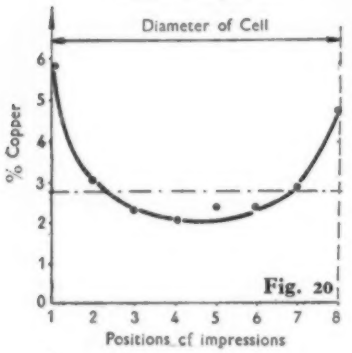


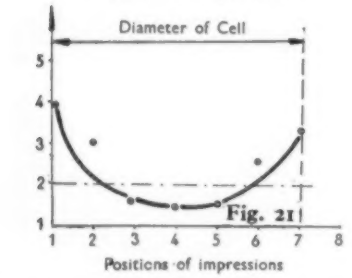
External zone. × 500

Fig. 17.—Micro-hardness tests on an aluminium—4% copper alloy, chill-cast ingot, "as-cast" condition, etched.





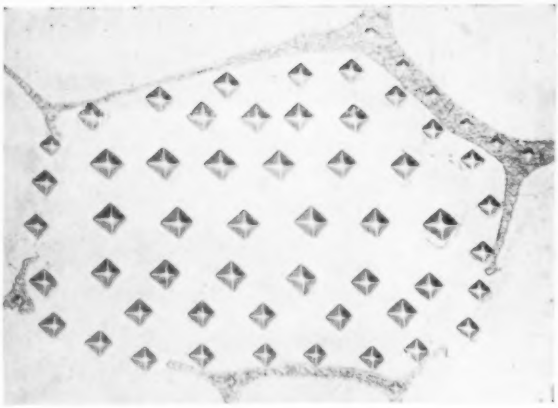




Figs. 19-21.—Distribution of the copper content across cells of a chill-cast aluminium—4% copper alloy ingot (external zone), determined by micro-hardness tests.

average hardness of the inner zone of the cell is 48 kg./mm.² (0·9% copper) and of the outer zone of the cell, 78 kg./mm.² (3·7% copper). Almost complete levelling of the concentration is attained after 9 hours treatment, as is shown in Fig. 26. The hardness values are 79 kg./mm.² (3·8% copper) and 78 kg./mm.² (3·7% copper) for the outer and inner zones, respectively.

After longer soaking periods the hardness still increases, since the $CuAl_2$ phase precipitated at the boundaries of the cells dissolves so slowly. After a soaking time of 68 hours, the hardness reached a level of about 90 kg./mm.² all over the cell, corresponding to a copper content of 4·5% (Fig. 27). By this time the whole area is almost homogeneous; only a few especially coarse $CuAl_2$ particles are distinguishable.



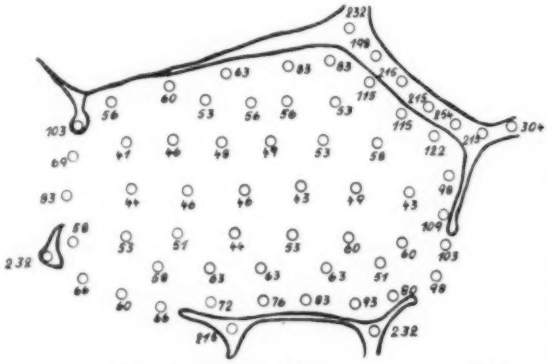


Fig. 18.—External zone. × 500

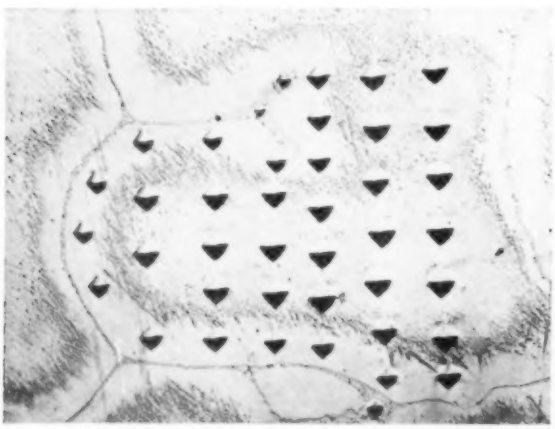
Aluminium-Copper-Magnesium Alloy — "As-Cast"

Fig. 28 shows a micrograph of a specimen from the core of the ingot (continuous casting); the average cell-diameter measured 65μ. The results of micro-hardness tests are given in Fig. 29 from which it will be seen that the average hardness of the inner zone of the cell is 51 kg./mm²; increasing to 80 kg./mm.² in the outer zone of the cell.*

Aluminium-Copper-Magnesium Alloy—Solution Heat-treated

One series of hardness tests was made as soon as possible after quenching the specimen, but there was always a delay of some hours before the polishing of the specimen was finished.

* As already mentioned, it is not possible to give any data on the chemical composition of the solid solution of this complex alloy on the basis of hardness numbers.



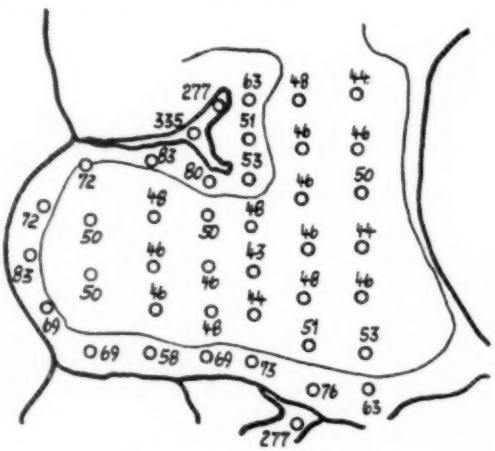


Fig. 22.—Core. × 500

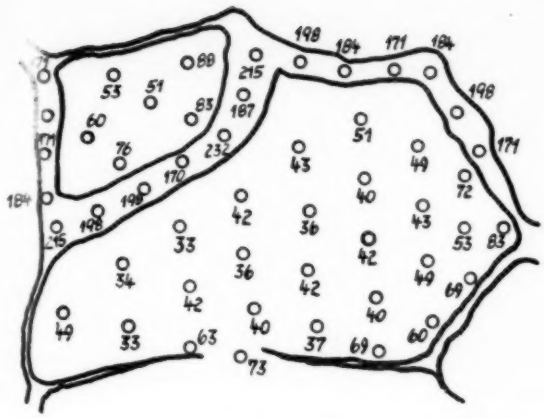
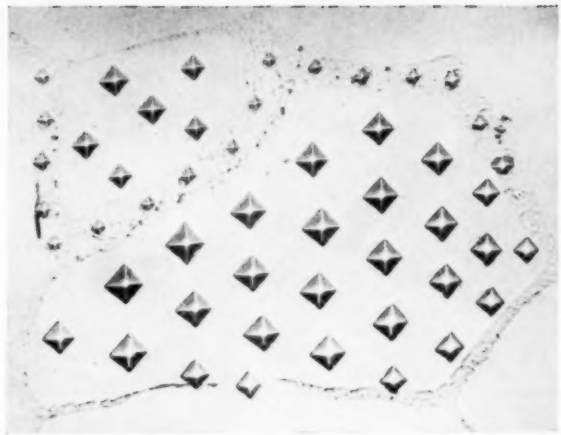
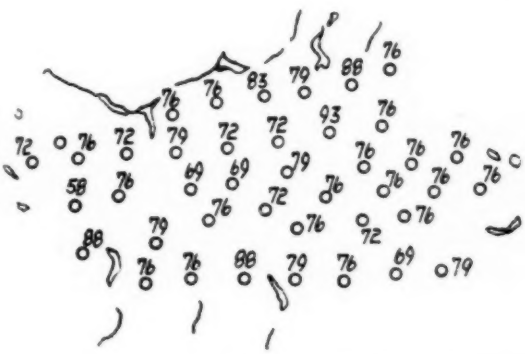


Fig. 23.—Core. × 500



Figs. 18, 22 and 23.—Micro-hardness tests of an aluminium—4% copper alloy chill-cast ingot, "as-cast" condition, etched.



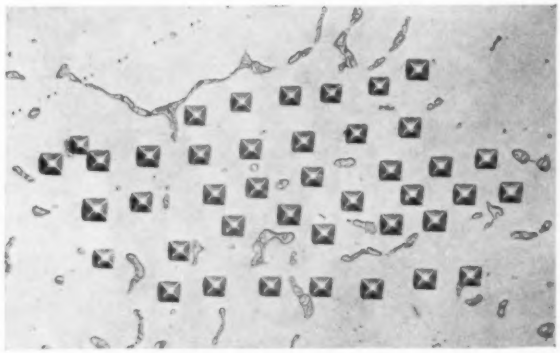
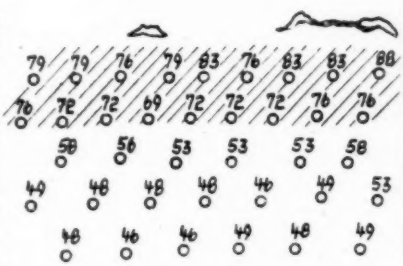


Fig. 24.—Continuous casting, soaked 20 mins. at 500° C. and quenched. × 500

In another series, the specimens were stored at room-temperature for 10 days, and then tested. It was expected that the first series would be almost free from age-hardening effects, whilst the second series would have higher hardness values after complete age-hardening at room temperature. The results of the first series showed such wide scatter that a reliable evaluation was not possible and, in consequence, only the results after complete age-hardening at room temperature are given in the following paragraph.

After soaking at 500° C. for 2 minutes, the differences in hardness within the cells still vary considerably, as shown in Fig. 30. The micro-segregation is not removed, but the average level is essentially higher than in the "as-cast" condition. The reason for this is that age-hardening occurs even with such a short soaking time. Fig. 31 is an example of an almost complete homogenisation after 3 hours soaking at 500° C. Within the whole grain, the hardness has reached



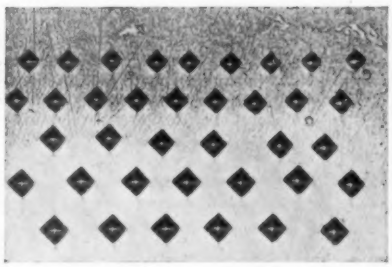


Fig. 25.—Chill casting, soaked 3 hrs. at 500° C. and quenched. × 500

an average value of 123 kg./mm.² No evidence of segregation is noticeable within the grain.

SUMMARY AND COMMENT

"As-Cast" Condition

The average results of the micro-hardness tests of the aluminium—4% copper alloy and the copper contents derived from them are set out in Table II. These results are average values obtained from numerous tests besides those shown in the illustrations.

TABLE II.—RESULTS OF THE INVESTIGATION OF SEGREGATION IN INGOTS OF ALUMINIUM —4% COPPER ALLOY—"AS-CAST" CONDITION

	Continuous Casting		Chill Casting	
	Outer zone	Core	Outer zone	Core
Micro-hardness (kg./mm.²)				
Outer zone of the cells	65	92	81	72
Inner zone of the cells	37	65	55	44
Copper-content (%)				
Outer zone of the cells	2·88	4·65	3·94	2·27
Inner zone of the cells	0·85	2·87	2·24	1·42
Micro-segregation* ..	2·03	1·78	1·70	0·85

* Difference in copper content between outer and inner zones of the cells.

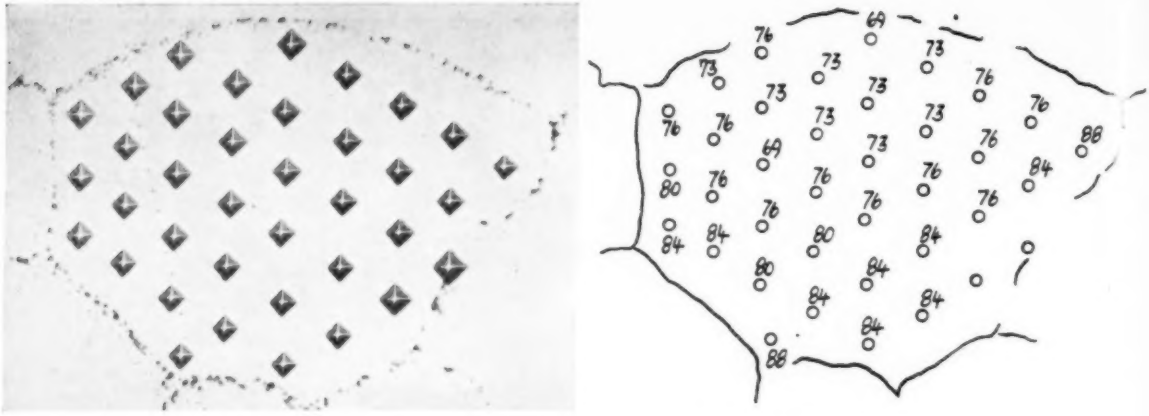


Fig. 26.—Chill casting, soaked 9 hrs. at 500° C. and quenched. × 500

Figs. 24-26.—Micro-hardness on an aluminium—4% copper alloy, continuous and chill-cast ingots, core, solution-heat-treated, etched.

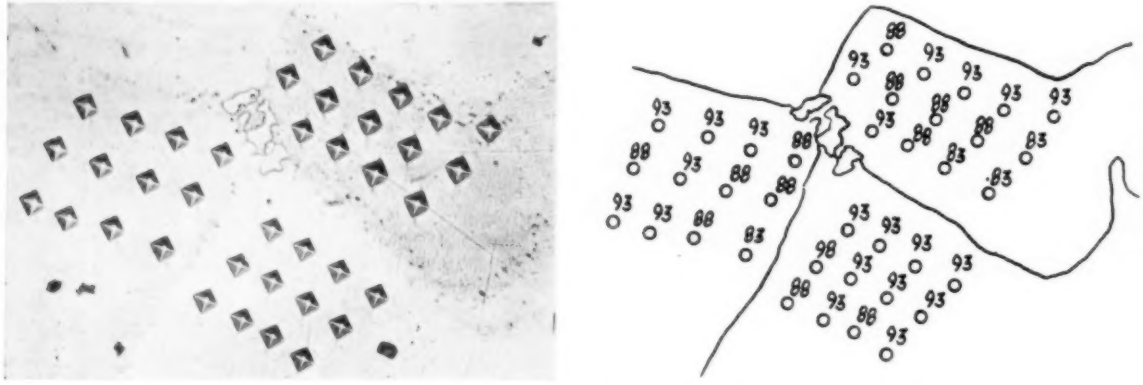


Fig. 27.—Chill casting, soaked 64 hrs. at 500° C. and quenched. × 500.
Micro-hardness tests on an aluminium—4% copper alloy, chill-cast ingot, core, solution heat-treated, etched.

From these results the following conclusions can be drawn.

Micro-segregation.—In both ingots, the amount of micro-segregation—i.e., the difference in concentration between the outer and inner zones of the cells, is greater in the outer zone of the ingot than in the core. In the chill-cast ingot the average micro-segregation is less than in the continuously cast ingot, probably as a consequence of the diffusion of copper during the slower cooling of the chill-cast ingot.

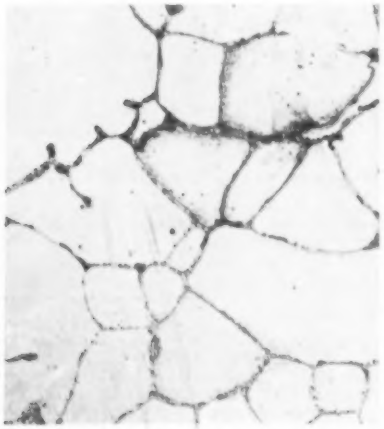


Fig. 28.—Photomicrograph on an aluminium-magnesium-copper alloy, continuously-cast ingot, "as-cast" condition. × 200

Macro-segregation.—In the continuously cast ingot, the average copper content of the cells in the outer zone is considerably lower than in the core. The primary dendrites would, therefore, give normal segregation, whilst zone-analysis usually shows inverse segregation in such ingots. The explanation of this apparent contradiction is that, during the growth of the dendrites from the surface of the ingot towards the core, normal segregation must occur. The nuclei first formed at the surface of the ingot must have the lowest copper content and, during the progress of solidification from the surface towards the core of the ingot, the liquid becomes slowly richer in copper. Nuclei formed after this copper enrichment in the centre must, therefore, be richer in copper than in the outer zones. Towards the end of solidification, the liquid penetrates from the core, through inter-dendritic channels, towards the outer zone and increases the average copper content of the material.[2] This excess of copper transferred to the outer zone by convection currents cannot be absorbed by the primary grains, but exists exclusively in the form of grain-boundary eutectic. The distribution of copper existing at the moment of solidification may be subsequently altered by diffusion. In the case of the continuous casting method the actual changes will be very small. Their magnitude can be estimated since the conditions of cooling are known in the case of the continuous

casting method. According to W. Roth,[8] the time required to cool from 600°–300° C., during which diffusion is possible, is about 15 seconds at the outer zone of an 300 mm. diameter ingot of an aluminium-copper-magnesium alloy, and about 200 seconds in the core. Since the depths of "sump" (liquid portion at the top) of ingots of different diameters are proportional to the square of the diameter, the cooling times for ingots of 200 mm. diameter, as used in this investigation, will be half those for 300 mm. ingots. It may be assumed that diffusion in the outer zone of the ingot is negligible, and only very slight in the core since the cooling time is not more than 2 minutes. As was shown in the investigation of the aluminium-copper-magnesium alloy, no appreciable change in the amount of micro-segregation occurs during a soaking time of 2 minutes. It is fairly certain, therefore, that the effect of diffusion can be neglected during the cooling of a continuous casting.

The conditions in the case of a chill casting are somewhat different. It can be assumed that, just as in the case of the continuous casting, the nuclei first formed in the outer zone of the ingot have a lower copper content than the core, since in this case, also, solidification starts at the surface of the ingot and proceeds towards the core, raising the copper content of the liquid. On account of the very slow cooling in the chill casting an appreciable levelling of the concentration between the eutectic at the boundary of the cell and the interior of the cell may take place by diffusion.

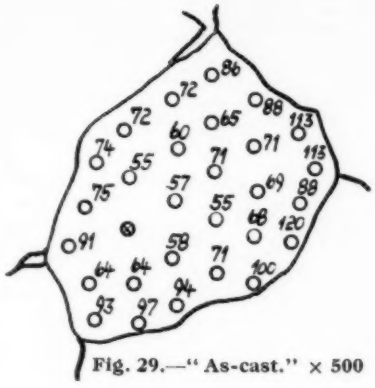
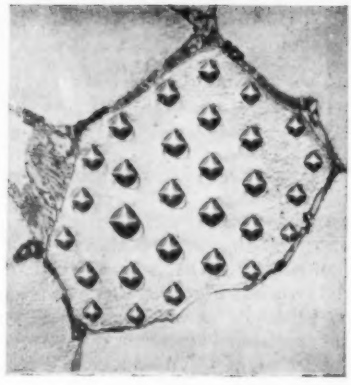
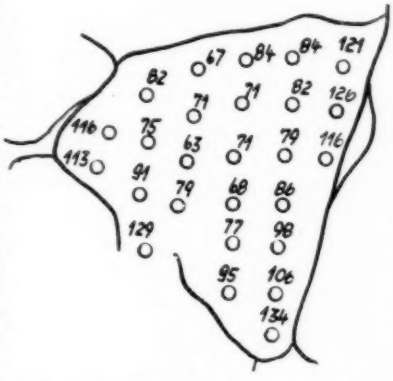


Fig. 29.—"As-cast." × 500





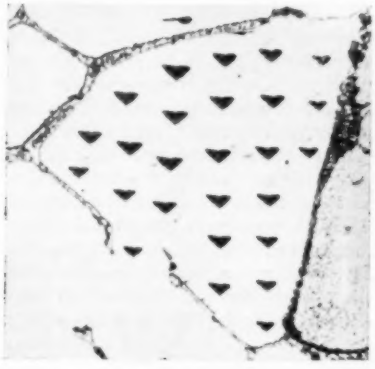


Fig. 30.—Soaked 2 mins. at 500° C., quenched and aged 10 days at room temperature. × 500

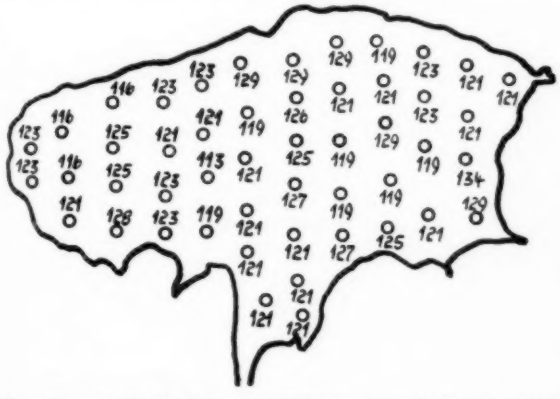
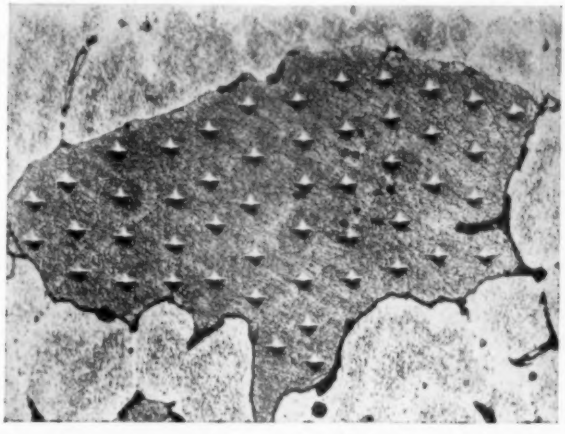


Fig. 31.—Soaked 3 hrs. at 500° C., quenched and aged 10 days at room temperature. × 500



Figs. 29-31.—Micro-hardness tests on an aluminium-magnesium-copper alloy, continuously-cast ingot, "as-cast" and solution heat-treated, etched.

TABLE III.—MICRO-HARDNESS (kg./mm.²) OF ALUMINIUM—4% COPPER ALLOY INGOT—SOLUTION HEAT-TREATED CONDITION

Heat-Treatment	Continuous Casting				Chill Casting			
	Outer Zone		Core		Outer Zone		Core	
	Outer zone of cells	Inner zone of cells	Outer zone of cells	Inner zone of cells	Outer zone of cells	Inner zone of cells	Outer zone of cells	Inner zone of cells
"As-cast"	68	37	88	58	82	55	72	44
20 min. at 500° C.	79	65	79	74	72	45	—	—
1 hr. " "	67	48	101	100	—	—	72	46
5 hr. " "	78	77	—	—	—	—	78	48
9 hr. " "	84	85	—	—	—	—	79	78
20 hr. " "	—	—	—	—	67	68	100	100
68 hr. " "	—	—	—	—	75	72	—	—

[8] Roth, W., *Aluminium*, **25** (1943), pp. 283–291.

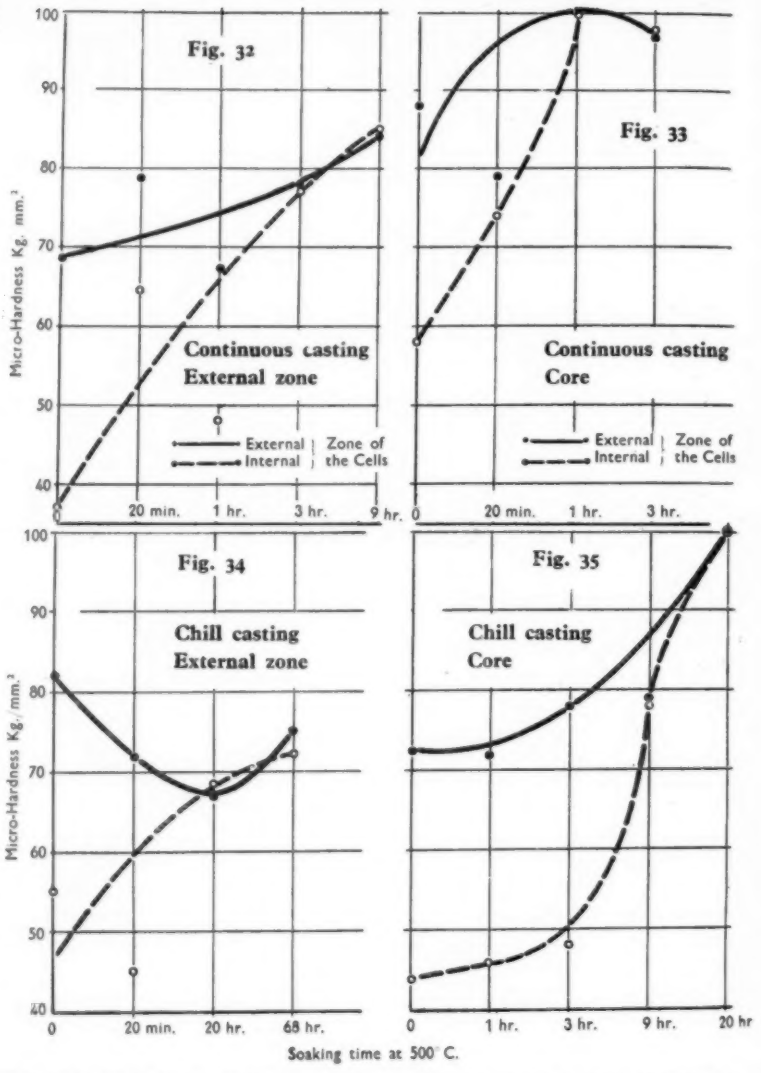
Solution Heat-treated Condition

The results of the tests on the solution-treated ingots of the aluminium—4% copper alloy are set out in Table III.

The results, which are shown graphically in Figs. 32–35, support the suggestion that diffusion plays an important part in these alloys. Due to the smaller cell-size and, therefore, the smaller diffusion paths, the homogenisation of a continuously cast ingot is completed in a much shorter time than that of a chill-cast ingot. The micro-segregation is practically removed after soaking at 500° C. for 3 hours in the case of the continuously cast ingot, and for 20 hours in the case of the chill-cast ingot. Macro-segregation, which is more pronounced in the chill-cast ingot cannot, of course, be removed by such a treatment.

The aluminium-copper-magnesium alloy, of which only a continuously-cast ingot was investigated, shows, in addition to the segregation effect, pronounced age-hardening effects. In Fig. 36, the hardness figures for both the inner and outer zones of the cells are plotted against the time of soaking at 500° C.

Fig. 37 shows the variation of hardness between the inner and outer zones of the cell, again varying with soaking time. It will be noted that the age-hardening effects have almost reached their maximum value after a soaking time of 10 minutes. After this time there is still a marked difference of hardness within the grains and even after longer soaking times there are still hardness differences between different grains, possibly resulting from variations in the average concentration in different parts of the specimen, corresponding to the quantity of copper-rich eutectic existing in these areas and slowly dissolved by a long homogenising treatment.



Figs. 32-35.—Effect of solution heat-treatment on the micro-hardness o aluminium—4% copper alloy ingots, continuous and chill-cast.

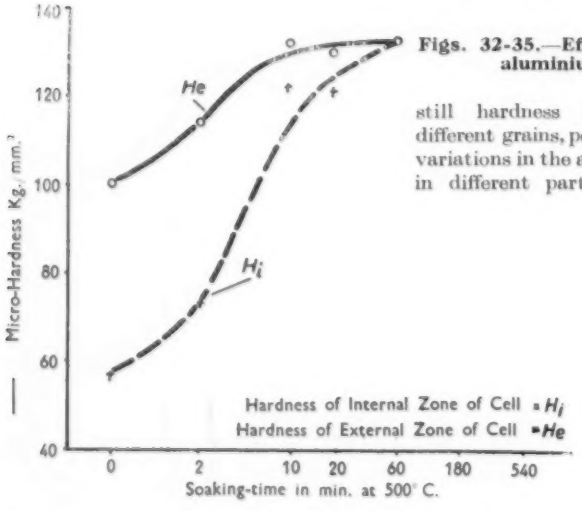

He
Hi
Micro-Hardness Kg. mm.²
Hardness of Internal Zone of Cell = Hi
Hardness of External Zone of Cell = He
Soaking-time in min. at 500° C.

Fig. 36.—Micro-hardness of the external and internal zones of the cells.

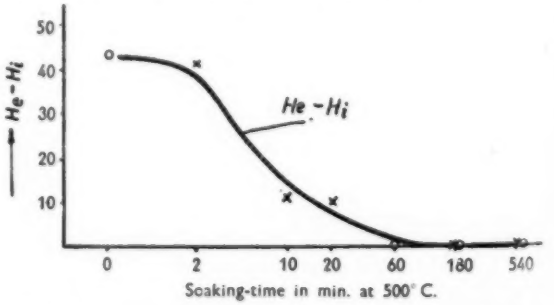


Fig. 37.—Differences in micro-hardness between the external and internal zones of the cells.

Creep and Fatigue Tests on Commercially Extruded Lead and Lead Alloy Pipes

By J. McKeown, D.Sc., M.I.Mech.E., F.I.M. and
L. M. T. Hopkin, B.Sc., A.R.S.M., A.I.M.

(*Communication from the British Non-Ferrous Metals Research Association.*)

Examination of service failures of lead and its alloys indicates the importance of a knowledge of the creep- and fatigue-resistance of these materials. The work recorded in this report was intended as a preliminary investigation to determine the degree of reproducibility to be expected from materials produced by extrusion on commercial presses, as it is well known that the creep properties, in particular, are very sensitive to variations in manufacturing conditions. It is concluded that work of a basic nature on the effect of alloying additions on the creep and fatigue properties cannot be carried out on extrusions made on commercial presses, where the many variables are not under sufficient control. The first part of this report was presented in our January issue.

The Effect of Cold Work on the Tadanac Lead from the Pipe Press

Two sets of tests at a stress of 750 lb./sq. in. were made on the Tadanac lead extruded on the pipe press. The cold work was applied by prestraining in tension at a straining rate of approximately 3 in./in./hour. The creep stress was calculated from the reduced cross-sectional area of a specimen after the prestraining and the creep was measured on a 4-in. gauge length marked on the specimen after prestrain. Each set of tests was made on specimens cut from the same 6 ft. length of pipe, one specimen from which was tested " as-received " to give a basis of comparison.

The first set of specimens was loaded immediately after 0, 1, 2, 3 and 5% prestrains. In the second set, the specimens were loaded immediately after 0, 6, 7·5 and 10% prestrain, and one specimen 24 hours after 10% prestrain. Also in this set two specimens were pre-strained 7·5% and 10%, loaded immediately after the prestrain, and then unloaded after 24 hours and examined for grain size.

First Set (Fig. 11)

In this set—i.e., prestrains of 5% or less, pre-working reduced the extent of the first stage of the creep curve, and lowered the creep rate slightly. The curve for 1% prestrain showed the first stage of decreasing creep rate; it may be noted that 1% was less than the immediate extension obtained on loading a work-free specimen at a stress of 750 lb./sq. in.

Specimens prestrained 3 and 5% showed a slightly increasing creep rate during the early part of the tests. This was thought to be due to softening of the cold-worked grains occurring at a slightly greater rate than the increase of plastic deformation during the creep test.

Second Set (Fig. 12)

(i) *Creep Rate and Life.*—All three creep curves for materials loaded immediately after prestraining showed an initial short period during which there was practically no creep. This period was longest in the case of the specimens prestrained 6% (3 hours) and shortest for that prestrained 10% (less than ¼ hour). Following this period, possibly the incubation period before recrystallisation commenced, the creep rate became rapid for a time and then decreased, the specimen work-hardening in a similar manner to a normal work-free specimen. The rate of this rapid creep was most likely dependent on the rate at which the specimen recrystallised, being

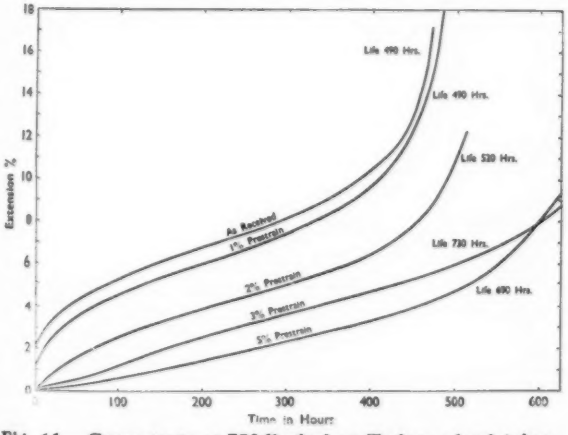


Fig. 11.—Creep tests at 750 lb./in.² on Tadanac lead (pipe press). Effect of pre-strain.

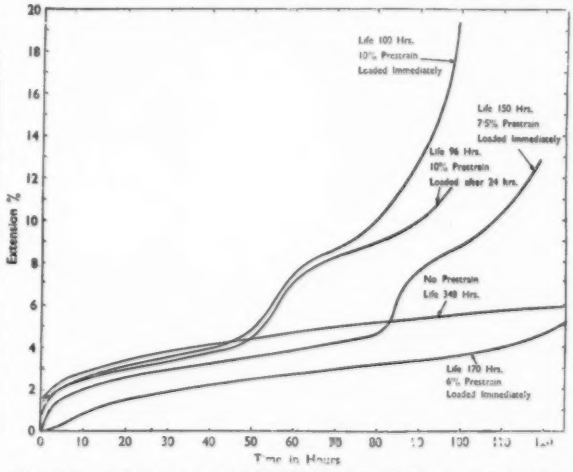


Fig. 12.—Creep tests at 750 lb./in.² on Tadanac lead (pipe press). Effect of pre-strain.

fastest after 10% and slowest after 6% prestrain. After about 5 hours testing time all specimens had approximately the same creep rate as the as-extruded material. The curves remained parallel until an inflection, which seemed to indicate a second recrystallisation, occurred at about 4% strain. In the case of the specimens prestrained 7·5 and 10% this large inflection was followed by a short period of decreasing creep rate, after which the rate again increased and continued to increase until failure occurred.

The specimen prestrained 6% showed a gradual increase of creep rate from 4% creep strain until failure. No necking occurred until well after the final turn up to failure. The lives of all these prestrained specimens were shorter than that of the work-free material.

The specimen prestrained 10% and left for 24 hours at room temperature before being loaded, had an immediate extension similar to a work-free specimen; this suggests that recrystallisation had occurred during the waiting period. The remainder of the creep curve was indentical with that for the 10% prestrained specimen loaded immediately.

The lives obtained in the two sets of tests on specimens loaded immediately were :—

	Prestrain %	Constant Rate of Creep Strain/hour at 750 lb./in.²	Life Hours
First Set	0	$1·35 \times 10^{-4}$	490
	1	$1·35 \times 10^{-4}$	490
	2	$1·18 \times 10^{-4}$	520
	3	$1·12 \times 10^{-4}$	730
	5	$0·95 \times 10^{-4}$	690
Second Set	0		348
	6	No definite constant rate of creep	170
	7·5		150
	10		100

In the first set the life increased to a maximum for the specimen prestrained 3% and then fell for a prestrain of 5%. The decrease in life with increasing prestrain in the second set of tests suggests that the reduction in life on increasing the prestrain from 3–5% is genuine, and it seems likely that, had it been possible to carry out all the tests using strictly comparable material, the life would have been a maximum for a specimen prestrained by about 3%.

(ii) *Grain Size.*—The "as-extruded" material had a mean grain area of 0·33 mm.². Specimens prestrained 10 and 7·5% and loaded immediately had, after 24 hours of creep, the same mean grain area, 0·16 mm.², as similarly prestrained specimens which had been allowed to recrystallise free of stress for 24 hours. X-ray examination of these two latter specimens showed that recrystallisation was not quite complete even after five weeks at atmospheric temperature. A specimen prestrained 10% and allowed to creep until just after the second inflection in the creep curve had a mean grain area of the order of 1 mm.², only slightly smaller than that found after failure.

In spite of the difference of 2 : 1 in the grain size of the "as-extruded" specimens and that which had been allowed to recrystallise for 24 hours after 10% prestrain, both creep curves ran parallel until the second inflection occurred. It must be remembered, however, that the recrystallised specimens were not completely work-free.

The Effect of Cold Work on 0.005% Ag + 0.005% Cu Alloy and 0.015% Tellurium Lead

Tensile prestrains up to 7% reduced the rate of creep of the 0·005% Ag + 0·005% Cu Alloy (see Fig. 13) and increased its life at a stress of 750 lb./sq. in.

% Prestrain	Life, Hours
0	2,770
1	3,000
3	3,500
7	4,100

The specimens prestrained 3 and 7% began to creep at a slow rate which increased gradually throughout the test and no truly constant rate was obtained—i.e., a curve of the Type 3 in the Greenwood and Worner Classification.

With the 0·015% tellurium lead a tensile prestrain of 10% reduced the minimum creep rate at a stress of 750 lb./sq. in. from $6·4 \times 10^{-5}$ strain/hour to $0·3 \times 10^{-5}$ strain/hour. After 1,300 hours the prestrained specimen started to extend rapidly so that it failed in the same time as the "as-extruded" specimen.

Effect of Heat Treatment on the Tadanac Lead from the Pipe Press

Five specimens, all from the same 6 ft. length, were

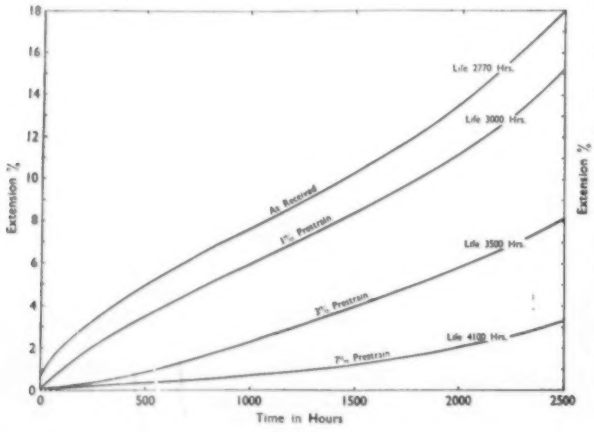


Fig. 13.—Creep tests at 750 lb. in.⁻² on 0.005% Ag. + 0.005% Cu alloy. Effect of pre-strain.

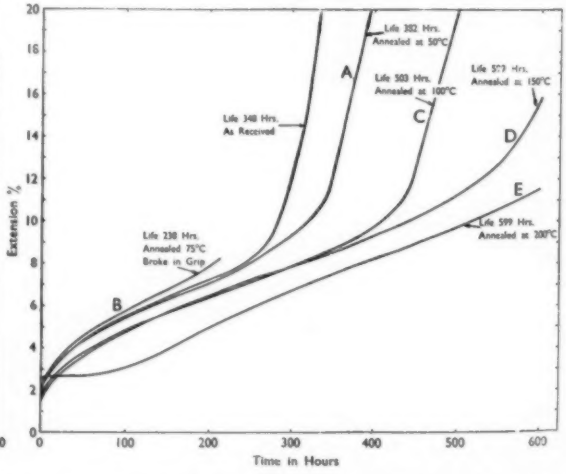


Fig. 14.—Creep tests at 750 lb./in.² on Tadanac lead (pipe press). Effect of annealing treatment prior to testing (24 hours treatment at each temperature).

tested in creep at 750 lb./sq. in. after treatments which produced the following grain sizes :—

Treatment	Mean Grain Area mm.²	Average Ratio Wall Thickness/Grain Diameter
A 24 hrs. at 50° C.	0·33	10
B " " 75° C.	0·33	10
C " " 100° C.	0·33	10
D " " 150° C.	2–3	3–4
E " " 200° C.	7–10	2

There was a notable similarity in the minimum creep rate of all the specimens (Fig. 14). The coincidence of the curves for specimens C and D, for the first 350 hours, was especially striking. It has been shown,[1] however, that the effect of grain size on the minimum creep rate of specimens of pure lead is small when the grain size is large, the minimum creep rate being most dependent on grain size when the grain size is small. The fact that the creep curve for specimen C left the minimum creep rate stage at a lower strain than that for specimen D, may again indicate that recrystallisation occurred more readily in a finer-grained material.

Specimen E, in contrast to all others, broke with a low general elongation (12·5%) and with an intercrystalline fracture. The creep curve for this specimen was somewhat similar to those obtained by A. A. Smith.[18] In Smith's tests the lead specimens had a grain diameter equal to the wall thickness and it seems possible that the high grain diameter/wall thickness ratio is the cause of this irregular type curve.

This type of curve was also obtained by Hirst[21] with a specimen consisting of five large crystals and with a single crystal. After the high initial rate of extension there was no creep during the period two to six days after loading, following which the normal work-hardening type of curve was obtained.

It is not possible from these limited tests to draw any conclusions as to the effect of grain diameter/wall thickness ratio on the life of a specimen.

FATIGUE TESTS

The fatigue testing of lead, especially in the form of pipe, introduces difficulties not experienced to the same degree with most other metals. Specimens must be machined without introducing unknown amounts of cold work, bending during handling and setting up the test must be avoided, the ends must be gripped in a manner which will not cause the lead to flow but will yet hold it tightly. Finally it is difficult to devise really satisfactory methods of measuring strains or stresses.

A machine for specific strain testing which minimised these difficulties is shown in Fig. 15. One end of the pipe specimen is gripped while the other is subjected to a rotating deflection produced by an eccentric chuck on an ordinary Wöhler machine. The specimen does not rotate and the centre ½ in. of its length is reduced in outer diameter to remove the position of maximum stress from the neighbourhood of the grip. A slight positive air pressure is kept in the specimen during test and, when cracking of the lead occurs due to fatigue, this pressure drops, thus breaking the contact with the mercury in the right-hand limb of the U-tube. The resulting interruption of a small electric current operates a relay and stops the testing machine. The deflection of the free end of the specimen is determined by an optical method which permits this measurement to be made during the actual progress of the test to an accuracy of $\pm 5 \times 10^{-5}$ in. The fatigue tests are carried out at a speed of 3,000 reversals per minute.

The bores of the pipes were prepared by a special broach which gave a fine finish with no trace of circumferential machining marks and left only a very thin worked layer. The outside of the specimen was turned using fine cuts with paraffin as a lubricant. Before testing, all specimens were degreased thoroughly inside and outside using trichlorethylene.

The endurance limit of any particular material was determined as the deflection corresponding to a life of 20×10^6 reversals of strain. In each material the endurance limit was determined using specimens machined from the one 6 ft. length.

In the early tests on grease-coated specimens it was found that considerable variation in the life of specimens was due to variation in thickness of the coating. The following procedure was adopted to obtain a coating evenly applied and reproducible in thickness inside and outside the specimen. Before being tested the degreased specimen was dipped into a standard solution of lanoline in petroleum ether, 1 : 5 by weight. On removal from the solution the ether evaporated quickly leaving a uniform coating of lanoline on the specimen.

Results of Tests

In both degreased and grease-coated conditions the highest endurance limits were given by the Alloy E and the 1% silver alloys. The improvement in the endurance limit resulting from the grease coating was much greater in the case of the pure leads and the 0·005% Ag + 0·005% Cu alloy than in the case of those materials

TABLE IV.—ENDURANCE LIMITS OF LEAD AND LEAD ALLOYS TESTED UNDER CONDITIONS OF SPECIFIC DEFLECTION

Material	Endurance Limit (20×10^6 cycles) Deflection at point X (Fig. 15) $\times 10^4$ in.	
	Degreased Specimens	Grease-coated Specimens
From pipe press :		
Tadanac lead	9	23
0·005% Ag + 0·005% Cu Alloy	10	26
0·06% Copper lead	14	20
Ternary Alloy No. 2	24	—
1% Silver lead	26	35
From continuous press :		
Tadanac lead	10	22
0·015% Tellurium lead	21	33
Alloy E	26	34

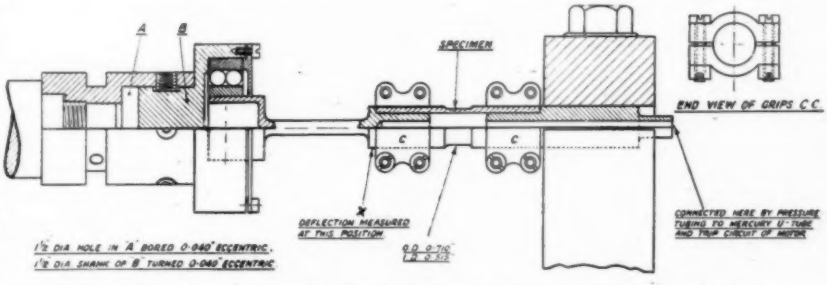

A
B
SPECIMEN
C
C
END VIEW OF GRIPS C.C.
DEFLECTION MEASURED AT THIS POSITION
CONNECTED HERE BY PRESSURE TUBING TO MERCURY U-TUBE AND TRIP CIRCUIT OF MOTOR
1½ DIA. HOLE IN 'A' BORED 0·040" ECCENTRIC.
1½ DIA. SHANK OF 'B' TURNED 0·040" ECCENTRIC

Fig. 15.—Machine for specific-strain fatigue tests on lead pipe specimens.

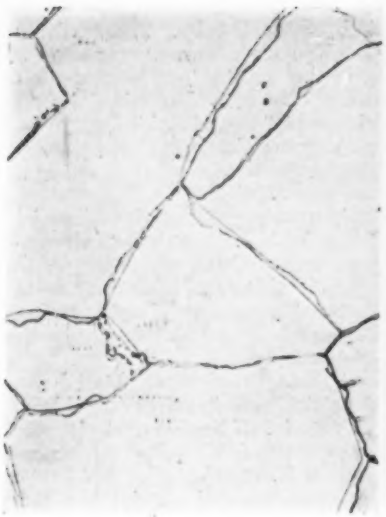


Fig. 16

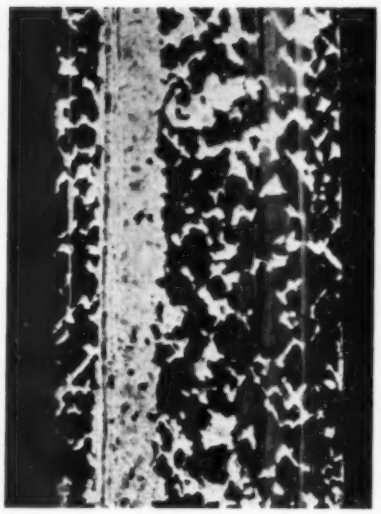


Fig. 17

Fig. 18

Fig. 16.—Discontinuous precipitation at the grain boundaries of the 0.015% tellurium alloy after severe fatiguing action. × 75. Fig. 17.—Longitudinal micro-specimen showing discontinuous precipitation chiefly associated with the major segregation lines in a specimen of Alloy E after failure at a stress of 2,000 lb./in.² × 12. Fig. 18.—Segregation lines which are continuous in undecomposed original grains appear as lines of precipitated particles in the areas of discontinuous precipitation. Specimen of Alloy E. × 200.

with higher alloy additions. Due to extreme lack of reproducibility of results, even in specimens taken from one 6 ft. length, it was not found possible to determine the figure for the endurance limit of the Ternary Alloy No. 2 in the grease-coated condition.

Examination of the fractured specimens showed that the cracking had commenced on the outside surface of the specimen and then penetrated the wall. This cracking was intercrystalline in all the materials when they were tested in the degreased condition.

Grease-coated specimens of the alloys also failed with a purely intercrystalline fracture, but the specimens of Tadanac lead from both types of press showed some transcrystalline cracking, although the crack which had caused the test to terminate was definitely intercrystalline.

Metallographic Examination of Failed Specimens

Examination of fatigue specimens after failure indicated that fatiguing action had less effect than creep on the metallographic structure. Discontinuous precipitation of the band type was observed at the grain boundaries of Alloy E and the 0·015% tellurium alloy specimens tested at high deflections in the grease-coated condition (Fig. 16). It was difficult to decide whether or not recrystallisation had occurred in the Tadanac lead specimens during test.

GENERAL DISCUSSION

As mentioned earlier, the object of the work described in this paper was to determine the degree of reproducibility to be expected from materials produced by extrusion on commercial presses and to obtain a comparison of some of the materials at present in use. In lead and lead alloys the creep properties are affected by change in grain size and by cold work, and it follows that the results obtained in the present work are strictly applicable only to the materials produced under the conditions described.

In spite of the care taken during the production of each material on the commercial presses, the reproducibility within each extrusion was poor for most materials and bad for Ternary Alloy No. 2. This poor reproducibility was undoubtedly due, in part, to lack of control of the extrusion conditions and, in part, to the heterogeneous microstructure of the extruded product. The observed lack of reproducibility must therefore be considered when comparing the relative merits of the materials in creep or in fatigue.

No evidence has been obtained to show that discontinuous precipitation has any effect on either creep or fatigue properties, but metallographic examination has shown that such precipitation occurs during test, and it is felt that this aspect of the subject warrants further study.

It is clear that work of a basic nature on the effect of alloying additions on the creep and fatigue properties of lead cannot be carried out on extrusions made on commercial presses, where the many variables are not under sufficient control. In view of this the investigations now in progress in the Research Association's laboratories are being carried out on materials produced on an experimental press in which all the known variables are under that close control possible in the laboratory.

ACKNOWLEDGMENTS

The authors wish to thank the Director and Council of the British Non-Ferrous Metals Research Association for permission to publish this paper. They also wish to acknowledge the assistance given by Mr. G. A. Dalziel, B.Sc., A.C.G.I. during the earlier part of the work.

APPENDIX

Temperature Limit of Solid Solubility

An attempt was made to determine the approximate temperature limit of solubility of Alloy E and Ternary Alloy No. 2. Specimens of these alloys, both in the "as-extruded" condition and following homogenisation by annealing at 250° C. for 24 hours, were extensiveiy

worked at various temperatures and then examined under the microscope for precipitation. The specimens had to be worked as otherwise equilibrium would not have been obtained in a practicable period of time.

The method adopted was to heat specimens of pipe ¾ in. long to the required annealing temperature, remove them from the furnace and deform them under a small drop hammer, then reinserting them into the furnace and holding them at the annealing temperature for a further period of 24 hours.

It was found that for Alloy E the limit of solid solubility was between 50° and 75° C. At 75° C. there was no precipitation in the specimen which had previously been homogenised at 250° C. for 24 hours, but the specimen tested in the "as-extruded" state showed precipitation in the locality of the segregation lines, although the rest of the structure was a homogeneous solid solution. Precipitation was present in both homogenised and "as-extruded" specimens after annealing at 50° C. Further experiments on specimens of the "as-extruded" material, slightly prestrained before annealing at 75° C., helped to confirm that the major portion of the alloy was in solid solution at this temperature.

The limit of solid solubility in Ternary Alloy No. 2 could not be definitely determined but is probably greater than 75° C. Extensive working of the homogenised material under the drop hammer, at room temperature, did not produce definite precipitation although the structure was very mottled, which might have been due to fine particles of globular precipitate. The grain boundaries etched more easily than in annealed or "as-extruded" specimens which had not been worked, and there was considerable pitting within the grains. Hot working, followed by annealing the alloy at 50° and 75° C. produced little difference in structure. It had been seen that the precipitate in Alloy E was finely dispersed after such drastic working under the drop hammer, so that the precipitate in the Ternary Alloy No. 2 might well have been sub-microscopic. The advantage of the stain etching of the antimony-rich constituent in the former alloy was not present in the case of the latter, thus increasing the difficulty of detection of any precipitated phase.

Evidence that Extrusion Lines were Due to Segregation

The evidence of the metallographic examination of all the materials, during the course of the work reported, was conclusive in showing that the lines indicating the direction of the movement of the metal during extrusion were associated with the segregation of the impurities or the alloying elements for the following reasons :—

(*a*) In Alloy E, continuous precipitation was observed, after ageing at room temperature for several months, in the locality of the segregation lines only (Fig. 3).

(*b*) During annealing at 250° C. the segregation lines on all the materials, with the exception of those containing an insoluble phase, gradually became less distinct and finally disappeared within 24 hours. This suggests that diffusion was taking place as the material became homogeneous. The 0·015% tellurium alloy, when annealed for 1 hour at 300° C., showed considerable grain growth, except in the region of the segregation lines. It may be noted that a 0·05% tellurium alloy showed no grain growth after a similar heat treatment, suggesting that the major segregation lines, in the 0·015% tellurium alloy, might have contained as much as 0·05% tellurium.

(*c*) In the determination of the temperature limit of solid solubility, it was found that a specimen of "as-extruded" Alloy E, after working and annealing at 75° C., consisted of a homogeneous solid solution, except in the regions of the segregation lines where precipitation was still present.

(*d*) After cold-working specimens of Alloy E the discontinuous precipitation occurred more readily in the locality of the segregation lines (Fig. 17).

(*e*) In specimens of Alloy E in which discontinuous precipitation was well advanced, it was observed that a segregation line in the undecomposed original grains became a line of densely precipitated particles when it entered the areas of discontinuous precipitation (Fig. 18).

The use of the term "segregation lines" which is used in the main paper is therefore a more accurate term than "extrusion lines" to describe this phenomenon, which was probably due to the segregated structure of the cast material persisting during extrusion.

Costing and Modern Accounting Methods in the Metal Industries

III.—Compiling Prime Costs

By S. Howard Withey, F.Comm.A.

Continuing his series of articles on costing, the author deals with the compilation of prime costs, particular attention being paid to labour costs. Reference is made to cold-rolling and wire-drawing operations.

TO obtain the best results from the costing system, the figures should cover a reasonably long period, and the various items charged against any process should be clearly defined and capable of easy interpretation. The rulings for the cost books vary in individual cases according to the operations covered; when hollow brass tube billets are cast on sand cores, for example, the main headings for the classification of core shop costs may be for wages, fuel, binders, sand, etc., whereas wire mill costs may have to be analysed under such headings as drawing, annealing, cleaning, machinery and tool repairs, repairs to muffles, wages, power, stores and acids, etc.

Costs of labour, materials, stores and outside services which are directly chargeable against the product constitute the prime cost, and in order to arrive at the total cost it is necessary to add all direct expenditure incurred for the purpose of raising productivity, including

an adequate amount to cover depreciation in the capital value of furnaces and other equipment due to actual physical deterioration, obsolescence, or effluxion of time, as well as the cost of all safeguards against risks and casualties. In some cases, costs are assessed at the furnace spout; in other cases they are taken at the moulds—i.e., up to the time of pouring in the metal. Generally speaking, it is advisable that, in addition to the cost of manning and charging the furnaces, melting costs should include power and fuel (including all items of expense incurred in raising temperatures); the cost of refining (including ore, electrodes, etc.); the ladling costs (including additional labour); metal losses; and the cost of refractories and other items.

When compiling prime costs, a column should be provided for the insertion of the output, and these figures should correspond with those recorded in the Output Book and should appear against each line of metal cast. The difference between the metals charged into the furnaces and the weights of the castings obtained will represent the deficit, the percentage relation of which should be carefully watched.

In cold rolling mill prime costs, the metal sheets and strips, etc., are usually grouped under definite headings and the rates determined to cover rolling, annealing, shearing and other operations. If it is necessary to take a number of job costs, a period of at least three months should be covered in order to arrive at reliable average figures. By multiplying the output of each group by the appropriate rate per ton, as shown in a separate column of the prime cost book, the monetary value of the output can be shown in an adjacent column. In addition to productive labour and general wages, the following are some of the main headings under which cold-rolled metal mill prime costs are usually allocated: Labour; fuel; power; repairs to muffles (wages and materials); repairs to machinery and tools; stores; timber; water; lighting; raw material; sundries; works charges; and acids (for cleaning).

When hot-rolled copper plates and sheets are manufactured, the various items representing the prime cost may also be allocated over the output, and this method is often adopted where hammering, flanging and dishing of plates is carried out. In the case of tube mills and wire mills, however, a good deal of detailed calculation and proportioning is involved and the method of costing is usually different. In general, metal-manufacturing costs comprise a number of post-production items in addition to labour, materials, establishment and other charges, and the final process in the compilation of prime costs is the collation of the relative elements in respect of each item of finished production. Logically, the system of costing is a part of the accounting system; to treat it separately is to restrict its practical utility.

Labour Costs

In addition to manual work, the labour cost element should include the non-manual effort of those who direct and supervise the manufacturing operations, whether the basis is day-work, piece-work, or payment by results and whether the salaries of overlookers, supervisors and inspectors are paid weekly or monthly. The wages of workers will be shown on labour cost sheets and will usually include the wages paid to foremen and charge hands, whereas the salaries paid to those not manually engaged will be debited to an account kept in the firm's nominal ledger.

When piece-workers clock in, the cards should pass through the progress office for the purpose of allocation, and when this has been completed and checked the wages sheets can be compiled. The entries on the wages sheets will include the name and pay of each worker daily, and the time worked during the week will be analysed under appropriate headings in the wages book, the headings corresponding as far as possible with those under which the trade purchases have been classified in the purchases journal or bought day book. The work performed by each clerk should be signed for, and each worker should attend personally to receive his or her pay, special arrangements being made for those who cannot possibly attend.

Day-workers usually enter details of all work performed each day on a card, and this should be initialled by the foreman whose records should be sent to the office each day for purposes of comparison. Particulars such as names, rates of pay, number of hours worked, deductions, etc., should be carefully verified, and typical rulings for time cards, time sheets, and wages sheets are reproduced on page 225.

In the financial books the figures of gross wages would be debited to a productive wages account as direct weekly postings from the "Bank" column on the payments side of the general cash book, the firm's contribution to State insurance being debited to a separate account kept in the nominal ledger, whereas the cost accounting entries would take the form of debits to the various labour cost accounts kept in the cost ledger, these charges being made direct from the wages sheets. A separate account should be opened for each order, giving the dates, check numbers, hours and amounts, the weekly totals of the latter being transferred to a Prime Cost Book, and at the close of the accounting period a summary of all current orders should be compiled so that any difference between the total and the balance of the productive wages account can be traced and adjusted.

Apart from materials purchased on capital and revenue accounts, all outside purchases relating to production

WIRE MILL

	Tonnage	Labour			Repairs to Machines, etc. (Wages)	Repairs to Muffles (Wages)	Power	Stores
		Drawing	Annealing	Cleaning				
Blocks								
Machines								
Total								

TIME CARD

Worker's No. : ..... Name : ..... Date : ..... 19 .....

Job or Order No.	Description of Work	Time		Number of Hours	Signed
		From	To		

TIME SHEET

No. and Name of Worker	Thursday		Friday		Saturday		Monday		Tuesday		Wednesday		Total Hours for Week
	From	To	From	To	From	To	From	To	From	To	From	To	

WAGES SHEET

Check No. : ..... Name : ..... Rate : .....

Date	Job or Order No.	Time		Overtime		Items £ s. d.			Charged	Total Time for Day	Total Day Wages £ s. d.		

order requirements should pass through an analytical purchases journal from where the monthly or other periodical totals can be debited to the stock accounts. Such purchases will, of course, remain as stock until such time as they are requisitioned and allocated to production orders. Each account kept in the cost ledger should show the weights, numbers and quantities of materials given out, the price of each kind and description and the full invoiced cost.

Piece-rate wages paid to workers engaged in drawing different classes of tubes may be classified under such headings as drawing, shouldering, annealing, pickling, filing, sawing and pointing, etc., and the wages book should show particulars and sizes of the tubes and the labour cost week by week. The mill cards will give the work done by each worker each day, and the total amount of wages paid out each week should agree with the figure shown under the heading of Productive Wages in the tube mill costs.

In the compilation of wire mill prime costs, the wages paid to the wire drawers on the blocks should be distinguished from the wages paid to drawers on the machines, providing the separate outputs are available, and the power charges should also be separately compiled. The amounts to be charged for certain other elements in the prime costs, however, may have to be shown in the form of totals, as they cannot always be dissected and recorded separately. For purposes of costing, the rates employed for the calculations are often based on the cost of drawing the wire to the various gauges and do not always cover all the manufacturing operations involved, the other elements being allocated over the total tonnage or other basis of output. A good general pattern of ruling for the Wire Mill Prime Cost Book is indicated

PRIME COST BOOK

Fuel to Muffles	Bricks, etc.	Wire Bar	Acids	Dies, etc.	Timber	Water and Lighting		General and Works Charges		Total

on pages 224-225, the headings being capable of modification to satisfy any specific requirement

When features peculiar to the individual requirements of buyers are incorporated in metal products which, for the most part, are ordered in relatively small quantities and are difficult or impossible to standardise, the method of controlling the progress of each job may be much more difficult and complicated than is the case when metal goods of the same kind are being produced in large quantities, and the material lists would have to give more details. Abbreviations for specifying both labour and materials are now used by some manufacturers, and control sheets describing in full the drawing and item numbers and the materials required often have to be maintained. These documents may be either hand-written, typed, or mechanically produced, and it is usually a simple matter to abstract and copy the headings, customers' references, drawing and part numbers when material is requisitioned from the stores.

A good deal of copying and posting can be eliminated by recording all lines of information relating to prime costs on a unit card designed for use right through to the compilation of the final figures, with the added advantage that data which is correct at the start must be correct all the way. In some cases, the job cost sheets of materials consist of the original requisitions on which the materials were issued, attached to supporting sheets which have been suitably slotted, and when used in conjunction with punched card or visible index systems the full advantage is obtained. By the simple process of slotting at both ends, any document can be converted into a unit card which can be readily filed, and unless they are withdrawn in the proper manner such cards defy any attempts to dislodge them.

A number of items are usually apportioned over the productive wages, and probably the most important of these is depreciation. Various methods of determining depreciation of fixed assets are adopted, and the next article will indicate how a physical control can be exercised over machines and plant and will show the correct method of recording items of capital outlay.

A New Method of Factory Lighting

An experimental installation of 2·5-kW lamps forms a novel and interesting solution to one of the most difficult problems of factory lighting that are usually classified as high bay lighting. The difficulties are principally because of the need to erect large pieces of machinery, and to have free travel for the necessary cranes and gantries and the height above the floor free from any obstruction (and the lighting fittings are obstructions) must be as great as possible—50 ft. or more. Yet at the same time the illumination at floor level must be adequate and uniform, the vertical illumination on machines under erection must be satisfactory and the illumination under any side galleries must blend reasonably well with the main lighting. In addition to this, a well-known pitfall awaits the illuminating engineer, that of service.

If service facilities for lamp replacement and cleaning are not given equal thought, the brightest design soon deteriorates to a third-rate effort because the factory management finds itself in a most uneconomical and impracticable position when contemplating the servicing of the equipment.

It is thought that all these points have received proper attention in the installation now in operation in a new BTH factory, the lighting equipment for which has been designed by the BTH Research Laboratory in co-operation with the factory engineers. The area to be illuminated measures 500 × 90 ft. and the mounting height for each fitting is 62 ft. Each lighting point consists of one 2½-kW mercury vapour lamp and two 1½-kW tungsten lamps. The illumination at floor level averages 11-ft. candles. Built-in cat-walks enable each fitting to be approached with safety, and the fittings are reversible through 180° so that servicing is done, not by overhead and uncomfortable working, but as if the fitting were on a bench. Under the galleries, illumination is provided by 80-watt fluorescent lamps and the colours of the two schemes blend so that there is little or no psychological disturbance.

The installation is a notable contribution to good working, both for the operatives on the factory floor, and for those who service it.

Machining Zinc Alloy Die Castings

The Zinc Alloy Die Casters' Association has recently published a book which provides a very useful illustrated guide to the various machining operations required in the finishing of zinc alloy die castings. Of course, one of the advantages associated with the use of these castings is the fact that machining is largely eliminated, but when it is necessary, in view of the long runs, it is important that it should be done in the most correct and efficient manner, and to this end the information given in this book is admirable. The book, which contains 70 pages, is profusely illustrated and excellently produced and to users and potential users of zinc alloy die castings it should be invaluable. For copies apply to the Association, Lincoln House, Turl Street, Oxford.

History of the Mushets

Mr. Fred M. Osborn, Chairman of Samuel Osborn & Co., Ltd., is engaged in writing a history of the Mushets: David Mushet (1772–1847) and Robert Forester Mushet (1811–1891), both distinguished metallurgists of their day, the former being the discoverer of the blackband ironstone of Scotland and the latter, among other things, being associated with Sir Henry Bessemer in perfecting his process and the discoverer of the high-carbon manganese-tungsten self- or air-hardening steel, the pioneer alloy steel. Mr. Osborn is very anxious to contact readers who have recollections of the Mushet era, in the period 1860–1900, or who have documents covering this period and bearing on the subject. It is believed that the Mushet family goes back to William the Conqueror's time, and any information about David Mushet's antecedents (he was born at Dalkeith in Scotland, his father being William Mushet who married Margaret Cochrane) would be appreciated.

The present generation has better ideas of the changes their fathers and grandfathers have seen and Mr. Osborn feels that it is important to keep alive memories and contributions of the past upon which much of our present progress is founded. We agree with Mr. Osborn's views and, if any readers can supply useful information on the subject, will they communicate direct with Mr. F. M. Osborn, Clyde Steel Works, Sheffield, 3.

LABORATORY METHODS
MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
INSTRUMENTS AND MATERIALS
FEBRUARY, 1950 Vol. XLI No. 244

Micro-radiography Applied to the Study of Iron Ores and Sinters

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Within recent years several papers have been published on the use of micro-radiography in the examination of steels, particularly in regard to micro-segregation of alloying elements. In the present article the author describes the application of the technique to the examination of iron ores and sinters for use in the blast furnace. This field is of considerable importance in the effort to achieve the most efficient and economic utilisation of raw materials.

MICRO-radiography is a relatively recent development of the technique of radiography for use in the study of textures and the distribution of materials on a microscopic scale. It has been applied successfully to the study of metals and alloys, concrete, etc., and its application to the investigation of iron ores and sinters is described here in the hope that its use may be extended in this and allied fields.

Radiography is already well established and widely used for the study of heterogeneity and defects in solids. X-rays are passed through the object and differences in absorption due to cracks, porosity, blowholes, segregations, etc., within the object can be either observed in the shadow image on a fluorescent screen, or recorded on a photographic plate in the form of a shadow photograph or radiograph. This technique is confined to the exploration of macroscopic features for the following reason.

From the point of view of absorption of X-rays a heterogeneous solid may be regarded as consisting of a great number of successive layers of material, each layer containing a mixture of grains of the various constituents of the solid. The layers are taken as orientated at right angles to the path of the X-rays, so that each ray has to pass through a heterogeneous column of grains. The absorption of the ray by such a column will be a compound effect, and, on average, will be much the same for all columns. The object as a whole will therefore produce a more or less uniform absorption and appear as a homogeneous substance. Only features such as cracks, segregations, inclusions, etc., which are large in proportion to the total thickness and affect a considerable number of these layers will affect the homogeneity of absorption and appear as lighter or darker areas in the shadow image.

In micro-radiography the technique is developed a step further. In order to reveal the fine texture and distribution of components in a heterogeneous object a small sample is taken and reduced to a thin slice, similar to the thin sections used for microscopic examination by transmitted light. If X-rays are passed through such a section, whose thickness is within the range of the average particle size of the object, a fairly sharp shadow image will be produced showing up the different grains due to the differences in their powers of absorption. Contrast in the shadowgraph can be increased by choosing the radiation so that it is absorbed particularly strongly by one of the constituents, the distribution of which, it is intended to study.

The amount of detail and sharpness of grain boundaries may be increased to some extent by reducing the thickness of the section to a figure below the grain size of the object. This reduces overlap of neighbouring particles at the grain boundaries and thus gives added sharpness of textural detail. There is, however, a limit to this thinning of the section because the difference in absorption between particles diminishes greatly with decreasing thickness, causing loss of contrast.

The shadow image is recorded on a photographic plate which is placed with its emulsion in direct contact with the specimen. The radiograph thus obtained can conveniently be examined under a microscope or an enlarged print may be produced from it. Direct enlargement of the shadow image, by placing the photographic plate at a distance from the thin section, was unsuccessful because of the divergent nature of the X-ray beam, and the pronounced scattering of the rays by the section, which caused a complete blurring of the image. Photographic details will be discussed more fully later in this article.

Field of Application

Micro-radiography, as outlined above, was first applied to ores by Kirchberg and Möller,[1] and the present author developed the technique for the study of two related problems, the distribution of iron in low grade British iron ores and in sinters. For some time it had been clear that more information was needed on micro-textures and distribution of material to supplement knowledge gained by customary and highly-developed methods of microscopy and chemical analysis. It was desirable not only to identify definitely the various iron-bearing components of ores, and to assess correctly their respective iron contents, but also to determine the distribution and textural relationship of iron-bearing and iron-free constituents, their amount and manner of intergrowth,

1 Kirchberg, H. and Möller, H., *Mitteilungen aus dem Kaiser Wilhelm Institut fuer Eisenforschung*, 1941, **23**, part 17.

respective particle size, and other details of texture. It is hoped to demonstrate the scope of micro-radiography in this respect.

The second problem concerns sinters. Modern mechanised quarrying methods result in a large proportion of finely broken material being included in the ores as supplied to the blast-furnace plants. Furthermore, concentration methods used with certain ores also result in a supply of rather fine-grained material. Such material must be reassembled into larger lumps before it can be fed into the blast furnaces under present-day practices, and one of the processes most widely used for this purpose is sintering.

The ore fines are suitably mixed with coke breeze and ignited under blast at a temperature high enough to cause a fritting and partial fusion of the material. Under correct conditions the sinter thus produced will contain most of its iron in the form of magnetite or hematite in a porous, easily penetrated matrix. Overheating will result in completely reduced free iron embedded in a heavily slagged matrix or, worse, result in the formation of iron silicates such as fayalite. Both these latter types of sinter give a very uneconomic rate of coke consumption in the blast furnace. There exists a definite need for a simple routine method of checking up on the day to day quality of sinter, and here again microradiography seems of great promise.

DETAILS OF TECHNIQUE

Sample Preparation and Mounting

The samples were prepared by the ordinary methods employed in making thin sections of rocks. Both ores and sinters were usually rather crumbly and had to be impregnated to prevent their disintegration during the final stages of grinding. Canada balsam was generally used for this purpose, but synthetic resins should be equally suitable. Briefly, a suitable lump, if possible free from cracks and other defects, was given a flat face of at least 1-in. square by cutting with a hacksaw or diamond-impregnated disc, or grinding with carborundum on an iron plate. The specimen was dried on a hot-plate, with some care in the case of ores, since overheating would have caused the break-up of hydrated minerals. The hot lump was then completely immersed in xylene and, after a few minutes soaking, returned to the hot-plate. Powdered Canada balsam was heaped on and pressed with a hot spatula into the face of the specimen, aided by the action of the solvent. The balsam usually penetrated the specimen to a depth of at least $\frac{1}{16}$ to $\frac{1}{8}$ in., so that, after allowing sufficient time for cooling and settling down, a slice about $\frac{1}{16}$ in. thick could be cut off without breaking. One side of this slice was ground perfectly flat and polished ready for mounting.

Mounting of the specimen differed from the usual petrographic procedure. The slice could not be mounted on an ordinary glass slide because, owing to requirements which will be discussed later, a type of X-radiation had to be used which could not penetrate such a thickness of glass within reasonable time. The specimen was, therefore, mounted on a thin cover glass which, for increased strength during the grinding process, was cemented to an ordinary slide glass. It was found to be most efficient to carry out the cementing of specimen to cover glass and cover glass to slide glass in one single operation, by simultaneously heating some Canada balsam to the required consistency on each glass. Final wet grinding was carried out in the customary manner with increasingly finer grades of abrasives on iron and glass plates. A final polish is not necessary for this technique, but some care should be taken to avoid a wedge-shaped section, and to obtain a reasonably smooth surface, because absorption of the X-rays is a direct function of the thickness of the section. Betteridge and Sharpe,[2] applying the technique to the study of steels, describe a mechanical device for obtaining uniformly thick sections. In brief, the device consists of an attachment to an ordinary metallurgical polishing machine. The sample holder is geared to the polishing wheel and is spring loaded; pressure is applied to the specimen centrally only by means of a pivot-like shaft which rests in a recess in the back of the plate to which the specimen is cemented. The authors evolved the device because they found hand operation tedious with hard steels, and because they experienced difficulties in obtaining uniformly thin sections. With the very much softer ores and sinters recourse to such a method was not found necessary by the present author.

The sections were generally ground to a thickness of about 30 microns so that they could also be studied in transmitted light under a microscope. Greater reduction in thickness usually resulted in serious loss of contrast in the radiographs, while slides thicker than 40–50 microns showed lack of detail due to overlap of grains. Grinding completed, the section is left uncovered and the mount is removed from the slide glass with a razor blade or by using a solvent.

For optical examination or photo-micrography the section can be mounted temporarily on a slide glass, using an immersion oil of suitable refractive index in order to cut down surface refraction. Kirchberg and Möller found it convenient to cement the mount permanently on to a slide glass which had a central hole about 8–10 mm. in diameter; the area of the section to be examined rested over the hole giving free access to the X-rays. This procedure gives good protection to the rather fragile mounts, but it was not used in the present work because radiographs of large areas were taken (1 sq. in. and more) and it would have been impracticable to use glasses with holes of such dimensions.

For exposure to the X-rays the section was placed with its uncovered side in contact with the emulsion of a photographic plate which had been cut to approximately $1 \times 1\frac{1}{2}$ in. The two were inserted in a flat container having a back of sheet brass and a front of opaque black paper through which the X-rays could enter without interference. (See Fig. 1). The paper front extends into a flap which can be tucked into the container after insertion of specimen and plate, this means of closing the container being sufficiently light proof and simple in handling. The container has a groove on either side so that it can be pushed into an adjustable holder mounted on rails. (See Fig. 2). The section can thus be located in any desired position and also moved along the X-ray beam to any distance from the tube window. A spring-loaded back and various other devices had been tried to ensure that the mount remained in close contact with the photographic plate inside the container, because even a small gap was found to cause lack of sharpness in the radiograph, but in the end it was found to be simplest and safest to fix the mount to the plate by means of two small plasticine pellets at opposite edges.

2 Betteridge, W. and Sharpe, R. S., *Journal of the Iron and Steel Institute*, 1948, **158**, part 2.

Nature of Ores and Sinters

Before going into details of X-ray technique a few notes on the nature and constitution of the ores and sinters are necessary.

The ores are essentially chemical precipitates of sedimentary origin and generally consist of heterogeneous concretionary grains, egg-shaped and up to 0·5 mm. in diameter, embedded together with fossil fragments and sand grains in a fine-grained crystalline or clayey groundmass. Iron occurs in the concretionary grains (ooliths), replacing the fossil fragments, and in the groundmass. Mineralogically some of the iron is present as carbonate (siderite), possibly contaminated by carbonates of magnesium and calcium. Then there are more or less complex hydrous iron-aluminium silicates which contain ferric as well as ferrous iron and perhaps also some magnesium ; chemically as well as mineralogically these are not at all well understood. The most common has been identified as chamosite. Finally, iron also occurs as various hydrated ferric oxides which are partly primary and partly represent weathering products of the other iron minerals. Iron-free minerals present include clay material (such as kaolinite) calcite, quartz, some phosphates of organic origin, and minor accessory minerals which are of no importance. For more detailed information the reader is referred to a most excellent description of these ores by J. H. Taylor.[3]

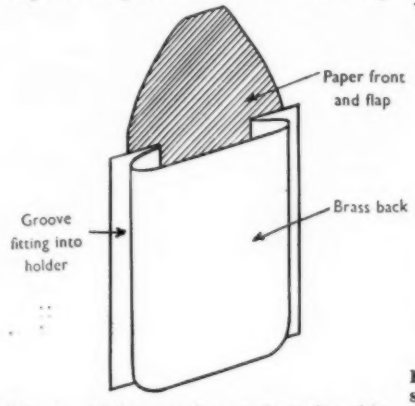

Fig. 1.—Light-proof container for thin section and photographic plate.

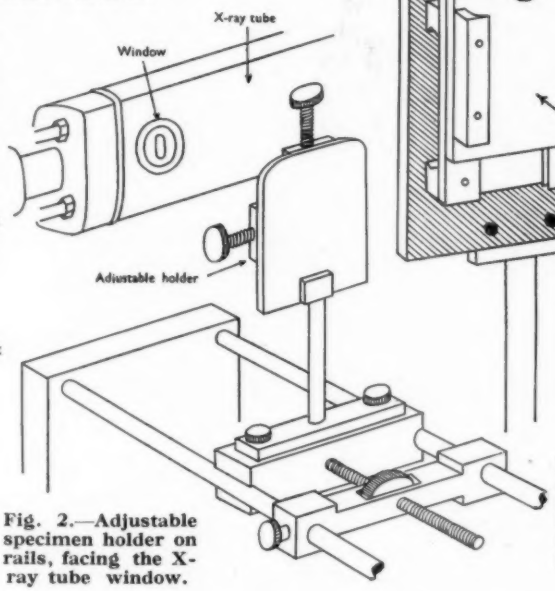

Fig. 2.—Adjustable specimen holder on rails, facing the X-ray tube window.

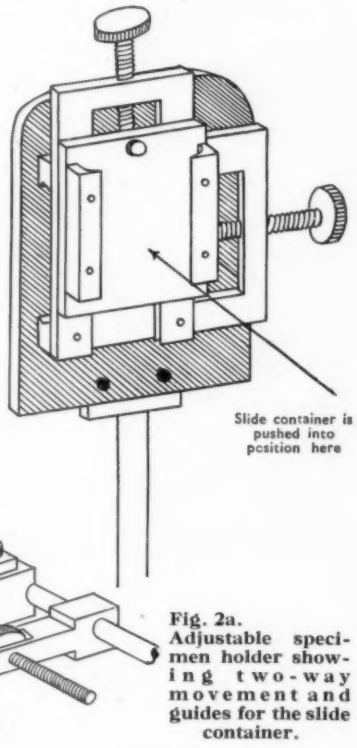

Fig. 2a. Adjustable specimen holder showing two-way movement and guides for the slide container.

Essentially then, we have a mixture of Fe, Al, Mg, Ca, Si, and a little P, apart from H, C and O, occurring in various forms of combination. This range also includes most elements which are likely to be found in the sinters. By choosing a type of radiation which is heavily absorbed by iron and relatively weakly absorbed by all other elements present, a good differentiation between iron and the rest should be possible. Furthermore, the intensity of absorption of X-rays by an element depends only on the amount of that element present and not on its valency or form of combination. If then a suitable radiation is used to produce a shadow photograph of an ore or sinter section, each grain in that section will appear lighter or darker in the radiograph entirely according to its iron content.

Radiography

Monochromatic radiation gives the best differentiation in absorption and the Kα rays of Ni and Cu are most suitable in this respect. Much detail on this point has already been published, for instance by Clark and Gross,[4] and detailed information will also be found in standard works on X-rays, such as that by A. Taylor.[5] By reference to Table I, it will be seen that the difference in power of absorption between iron and the other elements in question is greatest for nickel- and next best for copper-Kα radiation. Both these radiations lie close to the Kα absorption edge for iron, on the short wavelength side where absorption is strongest. A certain amount of Kβ rays as well as white radiation are probably always present, and in this connection some incidental benefit is derived from the interposition of the cover glass of the mount between the section and the source of X-rays. It has been found that the thin layer of glass exercises considerable absorption and probably acts as an efficient filter for all rays except the very much stronger Kα group. The use of additional filters did not produce any improvement in contrast or definition.

TABLE I.—LINEAR ABSORPTION COEFFICIENTS FOR Kα RADIATION
(Internationale Tabellen)

Absorber	Target Material				
	Mo	Cu	Ni	Co	Cr
Mg	7·6	70·5	83·1	105	208
Al	14·3	131	157	200	402
Si	15·6	140	176	216	446
S	20·5	186	227	285	557
Ca	30·3	263	321	395	777
Fe	301	2,550	3,120	470	903

3 Taylor, J. H., Petrology of the Northampton Sand Ironstone Formation. Memoir of the Geological Survey of Gt. Britain." (H.M. Stationery Office).

4 Clark, G. L. and Gross, S. T., *Industrial and Engineering Chemistry (Analytical)*, 1942, **14**.

5 Taylor, A., "An Introduction to X-ray Metallography." (Chapman & Hall, Ltd.).

The X-ray set, a Metropolitan-Vickers Raymax unit, was generally operated at 20 kV. and about 20–25 mA. As pointed out by Betteridge and Sharpe, the use of higher voltages would only increase the amount of shorter wavelength white radiation which would penetrate the section and produce general blackening of the photographic plate, with loss in contrast. For reasons not relevant to this paper the X-ray unit could not be operated at voltages below 20 kV. The characteristic Cu and Ni Kα radiations, however, are excited at about 8 kV. so that it might be possible to obtain a further improvement in the quality of the radiographs by using lower tube voltages, and higher currents for reducing exposure time. Kirchberg and Möller, for instance, used a copper target at 10 kV. and 30 mA.

The Raymax unit emits a rather narrow X-ray beam and in order to obtain a uniform exposure of the whole area of the section a distance of 6–7 in. was necessary between the tube window and the specimen. This, of course, prolonged the exposure time, and working with a beam of wider angle, shorter distance and shorter exposure time is much to be preferred. Self-radiation has not been investigated by the author, but its incitement in the iron of the section might be expected to cause reduction in contrast in the radiograph since it would compensate the effect of absorption of the primary rays. No obvious signs of such interference have been observed and self-radiation, if any, is assumed to be negligible.

Throughout the work Kodak Maximum Resolution plates were used with success and were found to be capable of considerable magnification without showing grain. Enlargements of up to 150 diameters have been made with remarkable preservation of detail. Under the conditions described above, exposure times of 10 minutes were generally required, but, as mentioned earlier, it is hoped to shorten exposure time considerably with smaller distance and increased tube current. Customary types of X-ray film were not suitable because of their double emulsion. The small distance between the specimen and the second emulsion is sufficient to cause serious blurring.

The plates were processed according to the makers recommendations. In practice it will be found that each worker can easily determine the optimum conditions for his particular needs, both in radiographic and photographic technique. Once these conditions are known they should be strictly standardised and not varied from exposure to exposure; this makes certain that any differences between radiographs are due to differences between the specimens examined.

It is advantageous to protect the emulsion of the radiograph with a cover slip cemented on with Canada balsam. By reducing the surface scatter of light, this gives the added benefit of increased clarity and transparency of the radiograph both for inspection and photography. The radiographs are normally viewed microscopically by transmitted light, and, wherever possible, are compared with the original section. With ores, at least, this comparison is necessary to give the full benefit of new information obtained from the radiograph. In the case of sinters comparison is usually difficult owing to the opaque nature of the sections, but some information can be obtained by the use of reflected light.

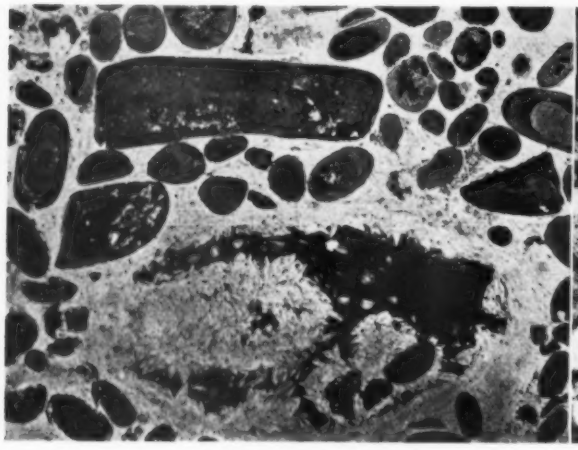

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Fig. 3.—Micro-radiograph. × 70

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Fig. 4.—Corresponding photomicrograph. × 70

A fossiliferous oolitic iron ore. The matrix is iron carbonate and the ooliths mainly hydrous iron-alumino-silicate. The bivalve shell in the lower part is completely replaced by iron carbonate.

EXAMPLES OF RESULTS

In illustration of the results obtained by the above method several radiographs are reproduced, some together with corresponding photomicrographs. The prints of the radiographs are negatives, an enlarged positive plate having been prepared from the contact images. Iron-rich material, therefore, appears white or light grey, iron-poor and iron-free material in darker shades of grey down to black. In clarity and amount of detail these prints fall short of the originals as seen under the microscope, but even so they demonstrate the new information and detail provided by the radiographs. The distribution of iron in the ores and differences in composition within individual grains are shown clearly. It also now becomes evident that, in thin sections of ores, the strong coloration of certain minerals, as well as staining and the presence of thin films of relatively opaque iron oxides, often gave an erroneous picture of iron distribution.

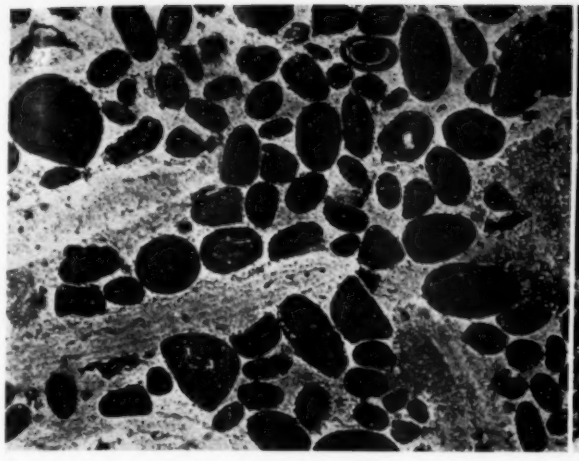

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Fig. 5.—Micro-radiograph. × 70

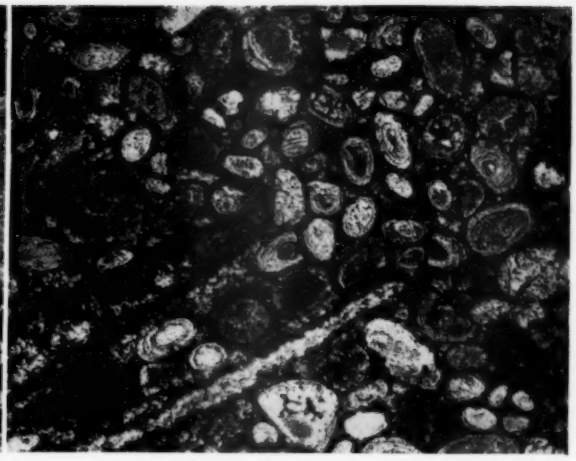

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Fig. 6.—Corresponding photomicrograph. × 70

An oolitic type similar to that in Fig. 3. showing, in the micro-radiograph, ooliths with concentric layers of different iron contents.

Figs. 3 and 4 illustrate the general similarity, and yet important differences, between the radiograph and photomicrograph of an ore section. The ore consists of ooliths of iron silicates, clay minerals, etc., and large fossil fragments embedded in a very fine-grained matrix which is mainly crystalline iron carbonate. The ooliths are generally low in iron content (20–30%) and the carbonate matrix high (about 48%). Most of the lower part of the field is occupied by a cross-section through a small fossil, a bivalve shell, showing the internal loop-like structure on which, during life, were suspended the respiratory organs. The whole of this shell has been replaced by iron carbonate (siderite) and a part of the internal cavity has become filled with siderite mud, new crystalline, and a few ooliths. Well developed rhomb-shaped crystals of iron carbonate protrude into the cavity of the shell and are clearly shown in the radiograph.

The concentric structure of an elongated oolith near the left-hand margin of the field is shown almost equally well in both pictures, but the photomicrograph provides no information as to distribution of iron, while the relative iron contents of the different layers could easily be determined photometrically from the radiograph. Irregular patches and occasional layers of iron-rich material (ferric hydroxide or ferrous carbonate) in some of the ooliths show up clearly in the radiograph. A particularly good example of this is seen in a very similar ore type in the next two illustrations (Figs. 5 and 6). An oolith near the upper margin of the field contains a layer of iron-rich material which shows up as an almost complete light ring in the radiograph. The rest of the grain is low in iron content and there is no difference between the inner and outer portions. By reference to the photomicrograph it will be seen that the outer and central zones appear conspicuously different in the thin section. The outer zone is coloured a deep reddish-brown wrongly suggesting a ferric oxide of much higher iron content than the light green silicate at the centre. The colour of the outer zone can be presumed to be due

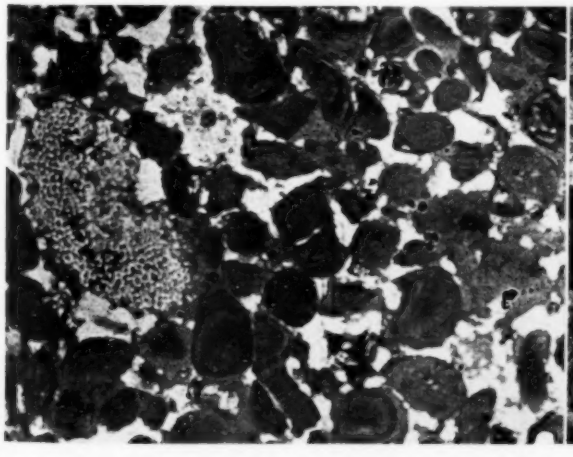

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Fig. 7.—Micro-radiograph. × 70.

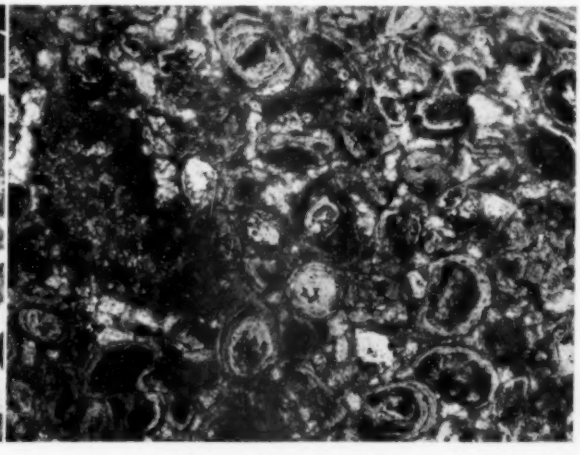

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Fig. 8.—Corresponding photomicrograph. × 70.

A certain lack of detail results from the use of a relatively thick section. This ore-type contains chamosite ooliths in a matrix of patches of siderite (light-rich in iron) and chamosite. The delicate structure of a fossil fragment is replaced by siderite, its pores being filled by chamosite.

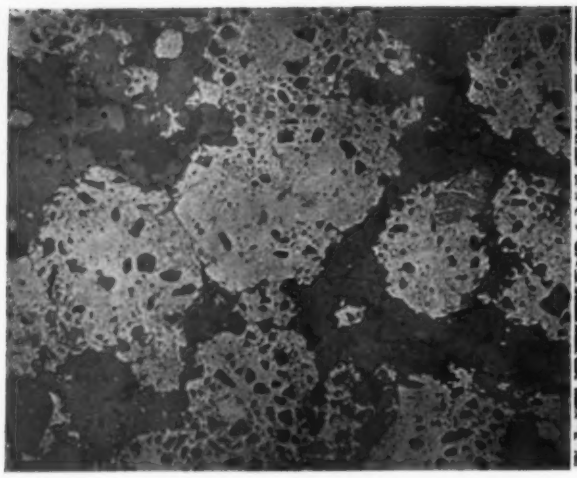

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Fig. 9.—Micro-radiograph. × 40.

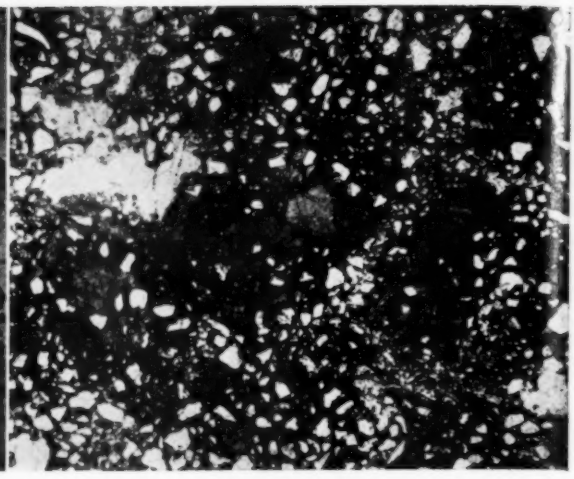

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Fig. 10.—Corresponding photomicrograph. × 40.

A chamosite mudstone with abundant angular quartz grains and nodules of secondary siderite (light). Owing to loss of detail during enlargement and reproduction, the quartz grains are hardly distinguishable from the chamosite and are more clearly seen in Fig. 10, in which, however, the siderite nodules can hardly be distinguished.

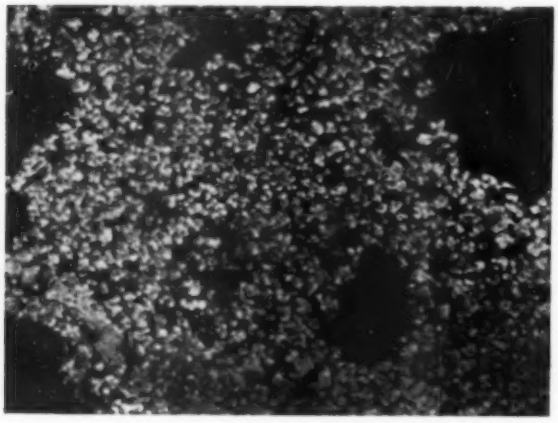

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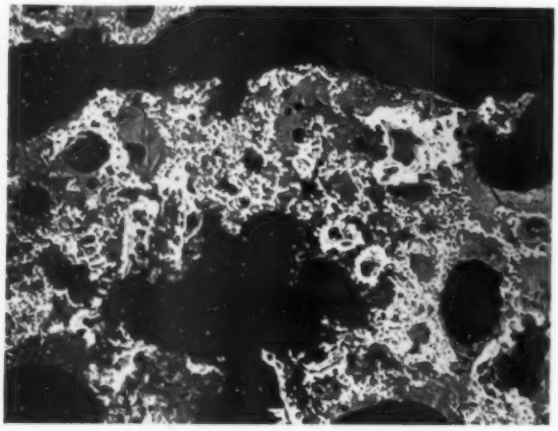
Fig. 11.—Micro-radiograph. × 123.

A good sinter, showing well-developed magnetite crystals in an iron-free matrix.

either to staining or to the presence of a thin oxide film. In the section the narrow iron-rich layer appeared almost opaque in transmitted light and reddish-brown in reflected light; it is probably a zone composed of ferric hydroxide. Further comparison of Figs. 5 and 6 will show other examples of such discrepancies between the X-ray and optical images.

The next two prints (Figs. 7 and 8) are reproduced mainly to show the effect of using a thick section. A considerable lack of detail is apparent as compared with the previous pictures, but the effect is worse in the photomicrograph. The radiograph clearly shows the replacement of a delicate fossil shell by iron carbonate (light), its pores being filled by iron silicate and clay. The groundmass of this ore is a rather patchy mixture of iron silicate and iron carbonate and its overall iron content is lower than that of the preceding ore type.

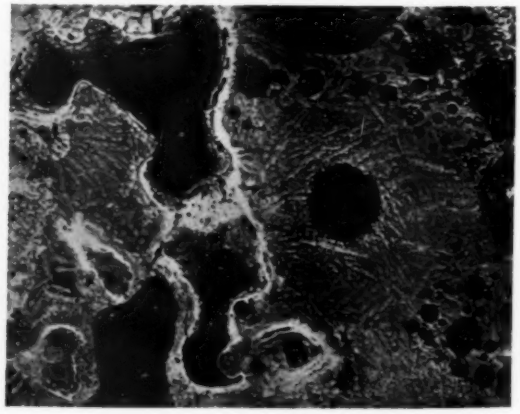
Figs. 9 and 10 show, respectively, a radiograph and photomicrograph of a rock containing abundant angular quartz grains embedded in a very fine-grained iron silicate matrix, and the whole invaded by a secondary



Reduced linearly in reproduction by a factor of approximately 2½

Fig. 12.—Micro-radiograph. × 70.

Overheated sinter showing blebs and stringers of free iron in a slagged iron-bearing matrix.



Reduced linearly in reproduction by a factor of approximately 2½

Fig. 13.—Micro-radiograph. × 70.

Overheated sinter showing layers of free iron, surrounding holes, and numerous lath-shaped crystals of fayalite.

growth of nodular (spherulitic) iron carbonate. The low iron content of the matrix, and the distribution and outline of the iron carbonate growths, are shown clearly in the radiograph whereas textural details are hardly visible in the photomicrograph.

The next three illustrations are intended to show the scope of the method in the field of sintering. Fig. 11 shows a radiograph of a sinter with an open-textured aggregate of well-developed minute magnetite crystals embedded in a porous iron-free matrix. Next, a badly overheated sinter in Fig. 12 with blebs and stringers of completely reduced free iron embedded in a slagged iron-bearing matrix. Finally in Fig. 13 a sinter which not only shows free iron in layers surrounding the holes and cavities, but also the formation of abundant lath-shaped crystals of iron olivine (fayalite).

The above brief examples are sufficient to show the usefulness of micro-radiography in the study of iron ores and sinters. It may be suggested that the method is equally adaptable to the investigation of many other types of ores, raw materials and, in fact, all heterogeneous solids, as long as, by choice of a suitable radiation, sufficient difference in absorption can be obtained to give a clear picture of the distribution of component materials.

Acknowledgment

The author wishes to record his indebtedness to members of the staff of the British Iron and Steel Research Association for advice and help given in the work, and to thank the Association for permission to publish this paper.

Book Review

MODERN METHODS FOR THE ANALYSIS OF ALUMINIUM ALLOYS

By a Committee of Chemists convened by ALAR. Editors: G. H. Osborn and W. Stross. pp. vii + 144, 11 illustrations. Chapman and Hall, London, 1949. Price, 13s. 6d.

Books which are produced as a co-operative effort often show clear indications of their divided authorship, but in this work the editing has been sufficiently skilful to retain the benefits accruing from the application of several counsels to a wise selection and presentation, with very little indication of lack of uniformity.

The methods chosen have been thoroughly tested in several laboratories, and the editors have wisely only included those methods which have a sound theoretical basis. The perpetuation of rule of thumb methods has been for many years one of the most serious errors of some branches of industrial analysis.

The lay-out chosen for the methods is very satisfactory—i.e., the principle of the method, the reagents required, the actual procedure, and the time taken in a determination. There is, perhaps, a certain unevenness in the statement of the principle. Thus, to take the first two estimations by way of example, the principle of the volumetric determination of copper, a classical procedure, is clearly and sufficiently stated; on the other hand, the paragraph under this heading for the estimation of magnesium by 8-hydroxyquinoline is merely an introductory generalisation—there is no equation, no indication of the formula of the complex, no explanation of the conditions of precipitation or of the removal of interfering ions. (In passing, too, it might be queried whether, in the statement of the principle for nickel, loss of nickel when removing manganese by ammoniacal bromine is due entirely to co-precipitation).

The first chapter of the book is an excellent introduction to that difficult topic, so often evaded, the method of sampling. Then follows a chapter dealing with the "classical," that is, with the volumetric and gravimetric methods for the elements normally present in aluminium alloys. It is noteworthy that methods are not presented here for either lead or titanium, so that instrumental methods must be chosen if it is desired to estimate these elements.

The chapter on electrolytic methods is well done, though there is perhaps a tendency to avoid theoretical principles. It is clear, however, that both the editors and the authors are up against a serious problem here, because of the difficulty of dealing adequately with the topic in a limited space. No quarrel can be found with the experimental methods.

In the next chapter, on absorptiometric methods, there is, rightly, a warning about the limitations of these methods, which is timely in view of the excessive enthusiasm sometimes shown for them. Indeed the principal criticism of the following chapter, on polarographic methods, is that a similar explicit statement of the limitations is not included. Isolated mention is made of certain disadvantages, but there is still room to stress the need for discrimination in applying this instrument to analytical problems.

The second section of the book deals with elements which occur less commonly in aluminium alloys, to wit, beryllium, bismuth, calcium, silver and sodium. Only one method is given for each element, the most appropriate type of procedure being chosen in each case.

The final section of the book, after a very fair statement of the arguments for and against, gives two composite schemes depending solely on absorptiometric methods.

The book is clearly a necessity for reference in any laboratory in which aluminium alloys are likely to be encountered, either as isolated or as routine analyses. It would probably be equally true to say that it should be available to all analytical chemists, since the lucid treatment of the methods should, in many instances, make their adaptation to other purposes a relatively simple matter.

C. L. Wilson

Prize-winning Photo-micrograph

Mr. Ronald Dawson, a member of the staff of the metallurgical laboratory of Raleigh Industries, Ltd., Nottingham, has been awarded a blue riband for a photo-micrograph exhibited in the metallographic section of the Exhibition and Congress of the American Society of Metals.

Mr. Dawson submitted a number of photo-micrographs and one of them was judged best in its class, entries for which were received not only from all over America, but from many other parts of the world.

The name of the Company hitherto known as Electronic Transmission Equipment, Ltd. has been changed to Mullard Equipment, Ltd.

An Improved Method for the Determination of Vanadium in Steel

By W. B. Shaw

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The direct titration methods ordinarily adopted for the determination of vanadium in steel are not entirely satisfactory, and although an indirect titration method was suggested more than thirty years ago, this too was not without its drawbacks. The application of the results of more recent research work to certain stages in the operation has resulted in a much more satisfactory method.

THE methods ordinarily employed for the determination of vanadium in steel are volumetric ones and usually depend upon the direct titration *either* of vanadyl compounds with permanganate *or* of vanadic compounds with ferrous ammonium sulphate.

The oxidation $V^{IV} \rightarrow V^{V}$ by permanganate in the cold is extremely sluggish and a titration based upon this reaction must obviously be far from ideal.

Acceleration of the oxidation by heating — advantageous in the case of pure vanadium solutions—is inadmissible in steel analysis, since a high temperature promotes a side-reaction between the ingoing permanganate and the chromium ordinarily present in the sample. Nevertheless, a procedure involving the direct measured oxidation of a vanadyl salt by permanganate in the cold is sometimes employed in ferrous analysis, often in the form of a combined method for the determination of Cr and V in the same sample of Cr-V steel.[1]

The reduction $V^{V} \rightarrow V^{IV}$ by ferrous ammonium sulphate proceeds much more rapidly and a method due to Willard and Young[2]—in which vanadate is converted into a vanadyl salt by measured reduction with ferrous ammonium sulphate—is widely used. That it is completely satisfactory will probably not be claimed, as the endpoint is not entirely free from uncertainty. A special pre-oxidised indicator is a necessity, since the use of plain diphenylamine sulphonic acid results in an indicator-error of appreciable magnitude. Even when the pre-oxidised indicator is used, a significant indicator correction must be applied to the titration results.

Indirect Titration

It is curious that the possibility of utilising the principle of indirect titration—that is, of forcing a reaction to completion by an excess of reagent and then determining the amount of reagent actually consumed by back-titration of the unused surplus—has been so little explored in connection with the determination of vanadium.

As far back as 1919, a rudimentary form of such a method was quoted by Macfarlane,[3] who obtained the details "from a memo. by J. Kent Smith, American Vanadium Co."

In Kent Smith's method, quadrivalent vanadium was first oxidised to vanadate by a visible excess of permanganate, in cold dilute sulphuric acid solution. The instructions continued: "just discharge this pink with N/20 ferrous sulphate. Note.—On the exact carrying out of this manipulation depends the accuracy of the vanadium determination." A measured (excess) amount of N/20 ferrous sulphate was then added—to reduce V^{V} to V^{IV}—and the unused surplus of ferrous sulphate was back-titrated with N/20 potassium dichromate, using ferricyanide as an external indicator.

This back-titration is possible because dichromate, unlike permanganate, does not oxidise vanadyl salts to vanadate. An accurate determination of the unconsumed excess of ferrous sulphate can therefore be made—by dichromate titration—without interference from the vanadyl compound present in the solution.

The weak link in this chain of operations is the determination of the exact point at which the excess of permanganate is to be regarded as having been removed by the ferrous sulphate. While this removal may be exactly judged in the case of a pure vanadium solution and might be observed with sufficient exactitude in the case of a plain vanadium steel, even approximate accuracy would seem to be impossible of attainment in the case of a steel containing only a moderate percentage of chromium.

In the presence of chromium, no sharp colour transition can be distinguished. Instead, the colour change, to the final green, passes through an unbroken sequence of intermediate tints, the location of the endpoint being most difficult to determine. This is, possibly, the reason why this method, so attractive in principle, should have received so little attention at the time of its publication.

It was hardly to be expected that Kent Smith could, in 1919, have brought this primitive scheme to any high degree of perfection. No satisfactory chemical means were then available for the quantitative removal, by selective reduction, of an excess of permanganate from a vanadate solution, since the Lang and Kurtz[4] plan—based upon successive additions of sodium nitrite and urea—was not published until 1933. Also, the discovery by Sarver and Kolthoff[5] of an internal indicator, really suitable for this titration, did not take place until 1931. In the absence of two such seemingly indispensable auxiliaries, it is difficult to see how further progress could have been made along this particular path.

Improved Method

It occurred to the author that an improved form of Kent Smith's indirect titration method—incorporating a recent modification[6] of Lang and Kurtz's selective

1 Hamner, H. L., *Met. and Chem. Eng.*, 1917, p. 206.
A.S.T.M. Standards, 1930, Part I, *Metals*, p. 359.

2 Willard and Young, *Ind. and Eng. Chem.* (*Anal. Ed.*), 1932, p. 187.
Willard and Young, *Ind. and Eng. Chem.* (*Anal. Ed.*), 1933, pp. 154, 158.

3 Macfarlane, W., "Practical Guide to Iron and Steel Works Analysis" (1919), p. 284.

4 Lang and Kurtz, *Z. anal. Chem.*, 1931, p. 288.

5 Kolthoff and Sarver, *J. Am. Chem. Soc.*, 1930, p. 4179.
Sarver and Kolthoff, *J. Am. Chem. Soc.*, 1931, pp. 2902, 2906.

6 Willard and Diehl, "Advanced Quantitative Analysis" (1943), p. 245.

reduction of excess permanganate and employing barium diphenyl-amine sulphonate[5] as internal indicator —might be free from the endpoint difficulties which seem to beset direct titrations of vanadium.

This expectation has been fully realised and it has been possible to work out rapid and accurate methods of determining vanadium, in both Cr-V and Cr-V-Mo-W-Co steels, which are wholly free from endpoint uncertainties.

The author has discarded urea, as nitrite eliminator, in favour of the more efficient sulphamic acid.[7] This substance possesses the great advantage of reacting instantaneously with nitrite, so that by its use the waiting period—so irksome when urea is employed—can be avoided.

The fact that sulphamic acid, $NH_2 \cdot SO_2 \cdot OH$, is a purely inorganic substance—of which the reaction products are only gaseous nitrogen, sulphuric acid and water—is a further advantage, since there is no longer any risk of interference with the endpoint by organic matter in the solution.

The reference sample used in testing the method devised for the determination of vanadium in Cr-V steel was British Chemical Standard Steel No. 224, having a stated vanadium content of 0·24% V. No difficulty was found in repeatedly obtaining results which either agreed exactly with this figure or only differed from it by not more than ± 0·01%.

In the case of the method worked out for the determination of vanadium in Cr-V-W high-speed steel, British Chemical Standard Steel "W.2," No. 167 (containing cobalt), was chosen. This steel has a stated mean vanadium content of 0·82% V. Again, no difficulty was found in obtaining groups of results which lay within the range 0·80–0·82%. For example, one group of eight estimations gave :—0·80, 0·80, 0·80, 0·81, 0·82, 0·82, 0·82, 0·82.

OUTLINE OF METHODS

Chrome-Vanadium Steel (Tungsten Absent)

The sample is dissolved in 20% sulphuric acid and the ferrous iron is oxidised by dropwise addition of conc. nitric acid to the hot solution until effervescence no longer takes place.

The solution is cooled, diluted and treated dropwise with 0·5% potassium permanganate, until a permanent pink coloration results.

The permanganate colour is destroyed with sodium nitrite and the surplus nitrite is decomposed by sulphamic acid. Barium diphenylamine sulphonate is added as indicator and an estimated excess of N/25·5 ferrous ammonium sulphate is run in.

The unused surplus of ferrous ammonium sulphate is back-titrated with N/25·5 potassium dichromate, and the volume of ferrous ammonium sulphate oxidised by the reduction of the vanadate is reckoned.

$$1 \text{ ml. N/25·5} \left\{ \begin{matrix} K_2Cr_2O_7 \\ \text{F.A.S.*} \end{matrix} \right\} = 0{\cdot}002 \text{ g. V.}$$

Chrome-Vanadium-Tungsten Steel

The sample is disintegrated by digestion with 30% sulphuric acid and sodium fluoride is added. Tungsten and carbides are taken into solution by oxidation with conc. nitric acid, according to the method of Willard and Young.[2] The solution is cooled and diluted. The determination is then completed as described above.

WORKING DETAILS

Chrome-Vanadium Steel (Tungsten Absent)

Dissolve a 2 g. sample in 75 ml. of Sulphuric-Phosphoric Mixture in a 650-ml. Phillips' beaker, with the aid of heat.

When completely dissolved, oxidise by the dropwise addition of conc. nitric acid to the boiling solution, until further additions cease to produce effervescence.

Cool down to room temperature and dilute to 200–250 ml. with cold water.

Spot in 0·5% potassium permanganate carefully, from a dropping bottle, until the solution remains pink. Avoid great excess (*B*).* Allow the pink solution to stand for 2–2½ minutes, to ensure complete oxidation of the vanadium (*C*). If fading takes place, restore the pink colour by further addition of permanganate.

Add 20–25 drops of 0·5% sodium nitrite, from a dropping bottle, and swirl. The permanganate colour will be discharged and the solution will turn green. Allow the green solution to stand for 15–20 seconds to ensure complete decomposition of the permanganate. Then add 5 ml. of 10% sulphamic acid and swirl to mix (*D*). Add 0·5–1 ml. of 0·2% barium diphenyl-amine sulphonate and run in, from a burette, an estimated excess of N/25·5 ferrous ammonium sulphate (*E*).

Back-titrate the unconsumed surplus of ferrous ammonium sulphate with N/25·5 potassium dichromate to a full violet endpoint.

Convert the ml. of ferrous ammonium sulphate into ml. of potassium dichromate and find the net ml. of ferrous ammonium sulphate consumed.

$$1 \text{ ml. N/25·5} \left\{ \begin{matrix} K_2Cr_2O_7 \\ \text{F.A.S.} \end{matrix} \right\} = 0{\cdot}002 \text{ g. V.}$$

On a 2 g. sample—

$$1 \text{ ml. N/25·5} \left\{ \begin{matrix} K_2Cr_2O_7 \\ \text{F.A.S.} \end{matrix} \right\} = 0{\cdot}1\% \text{ V.}$$

Chrome-Vanadium-Tungsten Steel

Transfer a 1 g. sample to a 650 ml. "Pyrex" Phillips' beaker and add 70 ml. of 30% sulphuric acid (*A*).

Bring to the boil. Cover the beaker with a watch glass and allow the liquid to digest—just at, or just below, boiling point—for 15 minutes (*F*). When digestion is over, remove the cover glass.

Add 5–6 g. sodium fluoride (weighed roughly) and swirl to mix. Bring the liquid to the boil and oxidise by the dropwise addition of conc. nitric acid, until further additions cease to produce effervescence. (About 30–40 drops of nitric acid may be needed). Boil for about 1 minute. A fairly clear green solution should be obtained. Add 150 ml. water (measured) and cool down to room temperature.

Spot in 0·5% potassium permanganate, from a dropping bottle, until the solution remains pink. Avoid great excess (*B*). (About 25–35 drops may be needed for 1% V). Allow the pink solution to stand for 2–2½ minutes (*C*) to ensure complete oxidation of the vanadium. If fading takes place, restore the pink colour by further additions of permanganate.

Add 20—25 drops of 0·5% sodium nitrite, from a dropping bottle, and swirl. The permanganate colour

7 Rosen and Ruchhoft, *Ind. and Eng. Chem.* (*Anal. Ed.*), 1941, p. 622.
Shapery and Gordon, *Ind. and Eng. Chem.* (*Ind. Ed.*), 1942, p. 792.
* Ferrous Ammonium Sulphate.

* Letters in parentheses refer to notes at the end of the article.

will be discharged and the solution will turn green. Allow the green solution to stand for 15–20 seconds, to ensure complete decomposition of the permanganate. Then add 5 ml. of 10% sulphamic acid and swirl to mix (*D*). Add 0·5–1 ml. of 0·2% barium diphenyl-amine sulphonate. A violet colour will slowly form. Run in, from a burette, N/25·5 ferrous ammonium sulphate until the violet colour has been completely discharged and then add 2–3 ml. in excess.

Back-titrate the unconsumed surplus of ferrous ammonium sulphate with N/25·5 potassium dichromate, to a full violet endpoint.

Convert the ml. of ferrous ammonium sulphate into ml. of $K_2Cr_2O_7$ and find the net ml. of ferrous ammonium sulphate consumed.

$$1 \text{ ml. N/25·5} \left\{\begin{matrix} K_2Cr_2O_7 \\ \text{F.A.S.} \end{matrix}\right\} = 0{\cdot}002 \text{ g. V.}$$

On a 1 *g. sample—*

$$1 \text{ ml. N/25·5} \left\{\begin{matrix} K_2Cr_2O_7 \\ \text{F.A.S.} \end{matrix}\right\} = 0{\cdot}2\% \text{ V.}$$

SOLUTIONS

20% *v/v sulphuric acid.*
30% *v/v sulphuric acid.*
Sulphuric—Phosphoric Mixture.
H_2SO_4 (conc.), 200 ml.; H_3PO_4 (syrupy), 150 ml.; Water *to* 1,000 ml.
0·5% *Potassium Permanganate.*
0·5% *Sodium Nitrite.*
10% *Sulphamic Acid.*
20% *v/v Phosphoric Acid.*
N/25·5 *Potassium Dichromate.*
1·920 g. " Analar " $K_2Cr_2O_7$ per litre.
0·2% *Barium Diphenyl-amine Sulphonate.*
N/25·5 *Ferrous Ammonium Sulphate.*
15·4 g. F.A.S. per litre. Dissolved in 5% v/v H_2SO_4.

Standardisation.—Withdraw 10·00 ml. from burette. Add about 5 ml. of 20% H_2SO_4, about 5 ml. of 20% H_3PO_4 and 3–4 drops of 0·2% barium diphenyl-amine sulphonate. Titrate to a full violet endpoint with N/25·5 $K_2Cr_2O_7$. (High H_2SO_4– acidity is necessary in order to secure a good endpoint). The ferrous ammonium sulphate solution should be checked every few hours (*H*).

NOTES

A. This large amount of acid is necessary in order to ensure accuracy. With lower acidity, results tend to be less concordant.

B. If too much potassium permanganate is added, extra nitrite may be required for its destruction.

C. If the solution is allowed to stand for a long time, the potassium permanganate may oxidise some chromium to chromate and so cause high results. There is little likelihood of significant interference from this source under 4–5 minutes.

D. If a number of determinations are being processed for titration together, they should be assembled at this point—i.e., after the addition of sulphamic acid.

E. 0·1% V = 1 ml. N/25·5 F.A.S.

F. Insufficient disintegration of the sample by sulphuric acid will prevent a clear solution being obtained on oxidation with nitric acid. Some steels are sufficiently disintegrated in a shorter time. The minimum time required by a particular material must be determined by experiment. Fifteen minutes seems to be a safe period for unknown samples.

G. Potassium dichromate can be purchased in a state of great purity and its quantitative solutions can be made up by direct weighing. If an independent check on the dichromate solution is desired—for example, to guard against the possibility of an error in weighing—samples of British Chemical Standard Steels (Nos. 224 or 167) can be included in a batch of vanadium determinations, whenever a fresh supply of N/25·5 potassium dichromate is brought into use.

As dichromate solutions appear to be completely stable for an indefinite period, no further check should be necessary.

H. Dilute solutions of ferrous ammonium sulphate are unstable and an N/25·5 solution cannot be relied upon to retain its strength unchanged for more than a few hours. Checks can conveniently be made while determinations are in progress, since the back-titrating solution also serves as the standardising solution.

The inconvenience caused by the rapid deterioration of the ferrous ammonium sulphate solution can be minimised by the following procedure: The factor of the ferrous ammonium sulphate solution is found by titration with potassium dichromate at the beginning of a run. If determinations are being carried out on steels containing 0·2–0·3% V, 5·00 ml. of ferrous ammonium sulphate solution provide a convenient excess for back-titration. Suppose that a factor of 0·965 has been found for the ferrous ammonium sulphate solution. Then: 5·00 ml., divided by 0·965, yield a volume of 5·18 ml., rounded off to 5·20 ml. 5·20 ml. of ferrous ammonium sulphate are therefore run into each determination, corresponding to an addition of 5·00 ml. of true N/25·5 ferrous ammonium sulphate. It is now only necessary to subtract from 5·00 ml. the actual ml. of potassium dichromate used in the back-titrations and to move the decimal points of the remainders one place to the left. The vanadium content is thus found, in the case of 2 g. samples, almost by inspection. This simple artifice does not seem to be as widely used as its convenience would appear to warrant.

Another insufficiently appreciated device is that of using volumetric solutions made up to correspond with some appropriate fraction of the equivalent of the active constituent.

Many vanadium determinations are carried out with N/40 ferrous ammonium sulphate (of which 1 ml. = 0·001275 g. V), thus involving a separate calculation in each case.

There seems to be no reason—other than unexamining conformity with tradition—why N/25·5 or N/51 solutions (having 1 ml. = 0·002 g. or 0·001 g. V, respectively), should not be substituted for N/40.

The exceptional convenience, in analytical repetition work, of solutions of equivalent-based, specific-purpose concentrations, appears to be overlooked by those whose experience has been mainly confined to the realm of general-purpose, round-number normalities.

Moreover, the influx of decimal normality notation into the field of industrial volumetric analysis seems likely to encourage, rather than to hinder, the adoption of cumbrous practices. The reason for the choice of, say, N/56 dichromate solution, for a particular iron estimation, may be obvious. The reason is hardly made more obvious—or the designation more convenient—by expressing this normality as 0·0179 N.

Primary Analytical Standards

By A. J. Nutten

Department of Chemistry, The University, Birmingham

Continuing the consideration of the various substances which have been advanced as primary analytical standards, the author deals with a further series of substances applicable to acidimetry and alkalimetry.

Sodium Bicarbonate

$NaHCO_3$. Eq. Wt. 84.015. Weak Base.
Indicator : Bromocresol Green.

North and Blakey[65] state that sodium bicarbonate is a satisfactory primary standard, but this has been contradicted by Lunge.[66] The substance has a higher dissociation pressure of carbon dioxide than the potassium salt, and this renders it less suitable than the latter compound as a standard substance.

Sodium Oxalate

$Na_2C_2O_4$. Eq. Wt. 67.01. ——→ Weak Base.
Indicator : Methyl Orange.

The use of sodium oxalate as a primary standard has been recommended by Sörensen.[67] His standardisation procedure is as follows :—

The oxalate is accurately weighed off and heated in a partly-covered platinum crucible for 15–30 minutes over a small gas flame, when conversion to carbonate results. From a burette is measured rather more acid than corresponds to the amount of oxalate. The cooled crucible is treated with a little water, and is placed upright in the beaker of acid along with the crucible lid. The beaker is then covered with a glass, and tilted to allow the acid to enter the crucible. The sodium carbonate is dissolved and most of the carbon dioxide expelled by heating on a water-bath. The solution is transferred to a flask, indicator added, the solution heated to boiling, cooled, and titrated with a standard sodium hydroxide solution.

The above procedure appears to be most unattractive and the recommendation of sodium oxalate as a convenient acidimetric standard is not justified.

Sym-Diphenylguanidine

$(C_6H_5.NH)_2C : NH$. Eq. Wt. 211.26. Weak Base.
Indicator : Bromophenol Blue.

Carlton[68] suggested diphenylguanidine as an acidimetric standard and lists some of its properties. Thus, though sparingly soluble in water, it is exceedingly soluble in ethanol. The product may be obtained analytically pure by three to four recrystallisations from toluene, and is unalterable in the air.

In his experimental study, Carlton used ethanol solutions of hydrochloric acid. The diphenylguanidine was dissolved in ethanol and bromophenol blue indicator added, the colour change at the end-point being blue ——→ yellow. If the solution being titrated was viewed against a yellow paper, the colour change was blue ——→colourless.

Using diphenylguanidine and sodium carbonate, Carlton obtained results which were in very close agreement. Thornton and Christ,[69] however, could not reproduce Carlton's results, and found that a discrepancy of 0·5–0·6% existed between the normality deduced from constant-boiling hydrochloric acid, and that obtained by titrating the basic substance.

They state that if purification of the diphenylguanidine could be accomplished it would be an excellent standard in neutralisation processes.

Young,[70] using diphenylguanidine purified by recrystallisation, obtained results comparing favourably with values obtained with sodium carbonate or borax. He titrated an ethanol solution of the base to *p*H 5·2 for N acid, and to *p*H 5·6 for 0·1N acid.

No further reference to the use of diphenylguanidine could be found in the literature, and the substance cannot be recommended as a standard till adequate purity tests are developed.

Thallous Carbonate

Tl_2CO_3. Eq. Wt. 234.40. Weak Base.
Indicator : Methyl Orange/Bromocresol Purple.

Hac and Kamen[71] recommended thallous carbonate as an acidimetric standard, while Berry[72] proposed it for the standardisation of both acids and potassium iodate.

Jensen and Nilssen[73] report a thorough investigation of thallous carbonate. They suggest that pure thallous carbonate is obtained only by recrystallisations in platinum vessels. They state that the compound is stable up to 150° C. at least, is non-hygroscopic up to 80% relative humidity, and does not absorb carbon dioxide from the air. A most suitable indicator is bromocresol purple, giving a sharp end-point in boiling solution.

The normality of hydrochloric acid was found by gravimetric determinations of the chloride, and by standardisations against sodium carbonate. The acid was then titrated against different samples of thallous carbonate, results obtained agreeing very closely in all cases, indicating the suitability of thallous carbonate as a primary standard.

Similar results by Hac and Kamen who used methyl orange as indicator showed that the titration values (111) obtained with thallous carbonate and hydrochloric acid agreed within a few thousandths of 1%, and were equally close to values obtained with sodium carbonate, borax and sodium oxalate as standards.

Guanidine Carbonate

$NH : C(NH_2)_2.H_2CO_3$. Eq. Wt. 60.55. Weak Base.
Indicator : Methyl Orange.

Dodd[74] claims that guanidine carbonate when prepared from calcium cyanamide is very pure, contains no

65 North, B. and Blakey, W., *J. Soc. Chem. Ind.*, **24**, 396 (1905).
66 Lunge, G., *Z. angew. Chem.*, **18**, 1520 (1905).
67 Sörensen, S. P. L., *Z. anal. Chem.*, **42**, 513 (1903).
68 Carlton, C. A., *J. Am. Chem. Soc.*, **44**, 1469 (1922).
69 Thornton, W. A. and Christ, C. L., *Ind. Eng. Chem.* (*Anal. Ed.*), **9**, 339 (1937).
70 Young, J. W., *Can. J. Research*, **B17**, 192 (1939).
71 Hac, R. and Kamen, K., *Collection Czechoslov. Chem. Commun.*, **4**, 145 (1932).
72 Berry, A. J., *Analyst*, **64**, 27 (1939).
73 Jensen, E. and Nilssen, B., *Ind. Eng. Chem.* (*Anal. Ed.*), **11**, 508 (1939).
74 Dodd, A. H., *J. Soc. Chem. Ind.*, **40**, 89T (1921).

water of hydration, and is not hygroscopic. It behaves as a mono-acid base when titrated against acid. He states that it is accurate enough for use as a standard in routine work. It has not, however, found much use in analysis, and is not readily obtainable.

Mercuric Oxide

HgO. Eq. Wt. 108.31. ——→ Strong Base.
Indicator : Phenolphthalein/Methyl Red.

Mercuric oxide can be readily obtained pure and dry, drying being carried out in a vacuum desiccator at room temperature. Exposure to sunlight must be avoided. When dissolved in excess of neutral potassium bromide or iodide solution (10·83 g. HgO + 200 g. KBr. in 300 ml. water, and dilution to 1 litre) a 0·1N potassium hydroxide solution results :—

$$HgO + 4KBr + H_2O \longrightarrow K_2HgBr_4 + 2KOH$$

and this can be titrated with acids in the presence of phenolphthalein.[75]

Kolthoff and van Berk[76] pointed out that, after proper corrections have been applied, the results are within 0·1% of the exact figure, but state that the method is not as accurate as standardisation of an acid against borax.

To carry out the standardisation, 1g. mercuric oxide and 20 g. neutral potassium bromide are dissolved in 25 ml. hot water, phenolphthalein added, and the solution titrated with acid till colourless. Methyl red is then added and the titration finished at the boiling temperature.

75 Rosenthaler, L. and Abelmann, A., *Z. anal. Chem.*, **53**, 608 (1914); *ibid*, **57**, 98 (1918).
Billmann, E. and Thaulow, K., *Bull. soc. chim.*, **29**, 587 (1921).
76 Kolthoff, I. M. and Berk, L. H. van, *Z. anal. Chem.*, **71**, 339 (1927).
77 Bieskei, J., *Z. anal. Chem.*, **88**, 414 (1932).
78 Kolthoff, I. M., *Pharm. Weekblad.*, **63**, 37 (1926).

Sodium Thiosulphate

$Na_2S_2O_3.5H_2O$. Eq. Wt. 248.19.
Indicator : Starch.

Sodium thiosulphate was recommended as an acidimetric standard by Bieskei.[77] His procedure for standardisation is as follows :—Pure sodium thiosulphate is accurately weighed out and dissolved in distilled water and the solution treated with an excess of potassium iodate and potassium iodide ; starch solution is added and the solution titrated with the acid to a permanent blue colour.

To standardise 0·1N acids, Bieskei used 2 ml. of 4% potassium iodate and 0·3–0·5 g. potassium iodide with 0·5 g. sodium thiosulphate. Very satisfactory results have been obtained.

Potassium Iodate

KIO_3. Eq. Wt. 35.67.
Indicator : Methyl Red/Methyl Yellow.

Kolthoff[78] has used potassium iodate for the standardisation of a strong acid. To standardise 0·1N hydrochloric acid he recommends as indicators methyl red, methyl yellow, bromocresol green or bromothymol blue.

The following reaction is utilised for standardising the acid :—

$$IO_3^- + 6H^+ + 5I^- \longrightarrow 3H_2O + 3I_2$$

For every 1g. potassium iodate, there should be allowed 1·5g. potassium iodide and 8g. sodium thiosulphate, though these need not be weighed out accurately. To prevent decomposition of the thiosulphate by local excesses of acid, the solution being titrated should be shaken thoroughly on addition of the titrant.

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The standardisation against potassium iodate is claimed to give results equally as accurate as those obtained using borax, sodium carbonate or sodium oxalate.

The substance appears to be satisfactory as a standard, but suffers from the disadvantage of a low equivalent weight.

Sodium Chloride

NaCl. Eq. Wt. 58.45. ——→ Weak Base.
Indicator : Methyl Orange.

Tananaeff[79] used sodium chloride for the standardisation of acids. A known amount of the salt is placed in a platinum dish and an excess of 1N oxalic acid solution added. The solution is evaporated to dryness and the residue ignited to sodium carbonate which is titrated in the usual way.

In a later paper[80] it was shown that the conversion of chloride to oxalate is quantitative to within 0·1–0·2%. The method, however, is of novel interest only and is not to be recommended.

79 Tananaeff, N. A., *Z. anorg. allgem. Chem.*, **154**, 186 (1926).
80 Tananaeff, N. A. and Lasarkewitsch, N. A., *Z. anal. Chem.*, **81**, 117 (1930).

Miscellaneous Microchemical Devices (XXIII)

Rotary Stirrers for Micro-Titration

By J. T. Stock and M. A. Fill (*Chemistry Dept., L.C.C., Norwood Technical College, London, S.E.*27)

PARTICULARLY when performing potentiometric or similar micro-titrations requiring insertion into the solution of electrode assemblies, etc., stirring by rotating the vessel is convenient in reducing congestion. The vacuum motor described below is useful for this purpose ; it is constructed from cheap and readily available material and is a development of the stirrers previously described.[1]

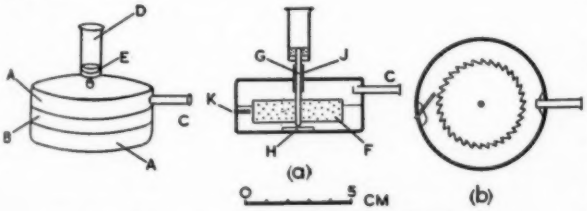

Fig. 1.—Stirrer unit. **Fig. 2.—Details of stirrer construction.**

The body of the device (Fig. 1) consists of two large (55 mm. inside diameter) bakelite caps *AA* as used for screw-top reagent bottles. When ready for assembly, the caps are placed mouth-to-mouth and are joined by bridging with a narrow strip of adhesive cellulose tape *B*. Connection to a water-jet vacuum pump is made at *C*. The semi-micro test tube containing the solution to be titrated is slipped into holder *D*. This is a 25-mm. length of glass tubing flared at the upper end and supported axially on the spindle by cork *E*, the joint being reinforced by sealing-wax.

Details of construction are shown at (*a*) in Fig. 2. Rotor *F* is a slice cut from an ordinary tapered cork and is about 45 mm. in diameter and 10 mm. thick. By means of a razor blade, about 32 ratchet-shaped teeth are cut in the periphery, as shown at (*b*). Spindle *G* is a darning needle and is thrust squarely and axially through the rotor, so that the point projects about 2 mm. When the device is assembled, the spindle point rests on bearing plate *H*, a small piece of microscope slide secured by sealing-wax in the lower half of the body. The upper half carries spindle bearing *J*, a short length of stout glass tubing squarely cemented in by sealing-wax. The tubing is selected so that the spindle fits snugly but rotates freely. Pump connection *C* and jet *K* are inserted in appropriately-drilled holes and likewise held by sealing-wax. Any convenient size of glass tubing may be used for the pump connection ; the jet is a piece of narrow melting-point capillary about 5 mm. long. To get the best results, the tangential position of the jet with respect to the teeth of the rotor should be adjusted by trial—rapid rotation should be obtained with mouth suction only. After connecting *C* to a water-jet pump *via* a safety bottle, suction should be adjusted to prevent excessively rapid rotation ; otherwise, the contents of the titration vessel may be lost through centrifugal action.

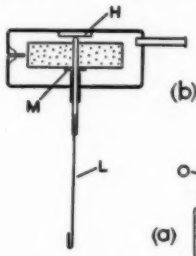

Fig. 3

Fig. 3.—Stirrer in use in inverted position.

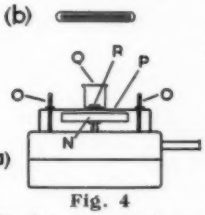

Fig. 4

Fig. 4.—Micro "magnetic bar" stirrer.

The device will also operate in an inverted position, thus enabling normal stirring (i.e., by insertion of a rotating stirrer into the sample) to be carried out. The stirrer head may conveniently consist of a length of No. 26 S.W.G. platinum wire attached to the projecting end of the spindle by winding a tight spiral, as shown at *L* in Fig. 3. When used in this way, it is desirable to place a small washer *M* of copper foil on the spindle between the rotor and the end of spindle bearing. This eliminates the sticking which may occur when the stirrer is out of use for some time. Although the rotor rests upon this washer when the stirrer is not in operation, application of suction to the pump connection causes the spindle to rise about 1 mm. until the point of the spindle encounters bearing plate *H*. Running is then nearly frictionless. For operation in this manner, the whole device may be made smaller, since a higher speed is permissible. Stirrers made from 25-mm. inside diameter bottle-caps and correspondingly smaller rotors have proved quite satisfactory.

A micro "magnetic bar" stirrer is shown at (*a*) in Fig. 4. Construction is generally as before, but the projecting end of the spindle is cut down and a small light powerful bar magnet *N* about 25 mm. long is attached symmetrically by sealing-wax, as shown. Two 8-B.A. brass bolts *OO* and appropriate nuts hold down platform *P*, which is another bottle-cap some 50 mm. in diameter. The platform should be rubbed down on glass paper both on the top and across the mouth, so that the top is flat and about 1 mm. thick, while the magnet is close to the underside when the apparatus is assembled.

A suitable titration vessel *Q* is a small specimen tube

1 Stock, J. T., and Fill, M. A., *Analyst*, 1949, **74**, 318.

cut down to a length of about 15 mm. Light grinding with fine carborundum paste is used to render the bottom flat. Stirrer-bar *R* is a 7-mm. length of sewing needle sealed in a thin-walled, snugly-fitting piece of melting-point capillary tubing. It is shown enlarged at (*b*).

In practice, the stirrer unit is kept running continuously. The sample is measured into the titration vessel, a stirrer-bar introduced, and the vessel placed centrally upon the platform. The stirrer-bar then follows the magnet round, and efficient stirring results. There is little tendency for splashing to occur, since the stirring action is most vigorous at the bottom of the sample.

The Work of the BTH Research Laboratory in 1949

A GENERAL review of the progress made in the B.T.H. Research Laboratory during the past year is the subject of an article by Mr. L. J. Davis, Director of Research, which will shortly appear in *B.T.H. Activities*. The immense field of interest of a research organisation associated with a large mechanical and electrical engineering concern will be evident from a study of the subjects dealt with, ranging as they do from metals to glass and from high voltage phenomena to street and factory lighting. In the limited space available it is only possible to refer briefly to a few items of direct interest to readers.

On the metal side, an item of outstanding interest to the electrical engineer is magnetic sheet steel which must have low watts loss, high permeability, good mechanical properties and low cost. It is important to have available, methods for the easy observation of grain orientation and, if possible, the rapid determination of percentage orientation, so that the results of the fundamental processes and the subsequent handling can be constantly checked. To this end two optical methods have been developed,* both using a polished specimen, etched by a special reagent which exposes facets parallel to cubic planes in the lattice. In the first method vertical illumination by polarised light is used, when facets in the plane of the specimen show no variation in intensity on rotation about a vertical axis. In the second method oblique illumination is used and, as the specimen is rotated, the angles at which various grains appear of maximum brightness are noted.

Work has also been carried out by observing the angular location of diffracted X-ray beams, to establish the orientation of individual grains in silicon iron.

There is no doubt that the carbon content has considerable influence on the properties of these materials and equipment has been developed for the determination of the very small percentages normally present.

Special welding problems are introduced in the construction of gas-turbine rotors and the effect of prolonged high-temperature treatment of such welds is being studied by determining the subsequent toughness and ductility.

The study of materials by the use of such methods as spectrography, infra-red spectroscopy, X-ray diffraction, etc., has resulted in the establishment of a service group for physical analysis. The services of the group are being increasingly called upon for the examination and identification of a very wide variety of materials, both metallic and non-metallic, used or encountered in the general work of the laboratory and of the company's factories. In most cases the methods are applied to minute traces of elements and to very small samples.

In the field of illumination engineering a novel solution to the difficult problem of high-bay lighting in factories has been found.† Interesting developments in light sources include a 1,000 W.–10 kW. mercury vapour lamp for high-speed cinematography—made possible by the characteristic which enables a mercury vapour lamp to be overrun for short periods by as much as ten times its normal loading, and a 250-watt compact source lamp which is sufficiently small to be interchangeable with the standard high-efficiency tungsten projection lamp.

In addition to the items mentioned, the article deals with insulating materials, glass and a range of scientific instruments and apparatus.

* See next column.

† See page 226

Optical Methods of Determining Grain Orientation in Electrical Sheet Steel

THE magnetic properties of sheet steel used in the construction of electrical equipment are dependent on the orientation of the grains with respect to the sheet. Two optical methods of determination have been developed in the B.T.H. Research Laboratory.

The first employs plane polarised light which illuminates at normal incidence the surface of the polished and etched polycrystalline specimen which is supported horizontally. An etching reagent is used which exposes facets parallel to cubic planes of the crystal lattice. The specimen is viewed in a vertical direction through a polaroid sheet crossed with respect to the polariser and it is rotated about a vertical axis. The grains are seen to fall into two classes, the first class comprising grains appearing always of minimum intensity and which do not change their appearance during rotation. These grains are reflecting the light after a single reflection at normal incidence. Since the reflecting facets are known to be cube planes, these crystals must also have a cube plane lying in the plane of polish. The second class consists of grains which exhibit variation from maximum to minimum of intensity as the specimen is rotated. These grains are reflecting the light after either double or triple reflection at oblique incidence. They have no cube plane near the plane of polish. The method is of greatest use where a certain orientation is known to be preferred; then the section to be examined can be chosen so that cube planes are expected and inspection shows whether this is indeed the case.

The second optical method also employs a polished specimen etched to expose cubic facets as before. The specimen is illuminated on the same horizontal microscope stage by light coming from one side only, the specimen is rotated and the angles at which various grains appear of maximum brightness are noted. From these data the orientation of each grain with respect to the sheet is calculated. A grain with a cube face in the plane of polish always appears dark. A grain with one cube edge in the plane of the sheet and the other two at 45° to the sheet, exhibits two maxima 180° apart, and a grain in which all three cube edges make nearly the same angle with the plane of polish exhibits three maxima.